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MANHATTAN DISTRICT HISTORY

BOOK V - ELECTROMAGNETIC PROJECT

VOLUME 8 - APPENDIX

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FOREWORD

This volume has been written to provide the reader with an understanding of the problems encountered and solved by research for the Electromagnetic Plant. It also furnishes factual research information on costs, organization and contract provisions. The text has been written as of 31 December 1946.

Most of the basic research was performed by Dr. E. O. Lawrence and his staff at the University of California. No attempt is made to detail the studies prior to 1 May 1943, but their extent is indicated by the status of the work on 1 May 1943 when the Manhattan District officially assumed direct responsibility for that work.

Each of the words and phrases which are defined in the Glossary, Appendix D, is designated, at its first appearance in the main text, by an asterisk (*).

The summary contains an abstract of every major subject treated in the main text of this volume, and paragraph designations in the summary refer to sections designated in the main text.

Other phases of the history of the Electromagnetic Plant are described in the following volumes:

Book V Volume 1	-	General Features
Book V Volume 3	-	Design
Book V Volume 4	-	Silver Program
Book V Volume 5	-	Construction
Book V Volume 6	-	Operations

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MANHATTAN DISTRICT HISTORY

BOOK V - ELECTROMAGNETIC PROJECT

VOLUME 2 - RESEARCH

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SUMMARY

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1. Introduction. The President of the United States, under the War Powers Acts, authorized research for the Electromagnetic Plant. The purpose of the Electromagnetic Research Program was to obtain in a short time the basic information needed to build and operate a plant for separating U-235. The scope of research has included the physical problems of mass spectrograph design and the chemical problems of preparing and recovering charge material. Most of the physical research and the original chemical work were performed at the University of California Radiation Laboratory (UCRL). Certain of the physical problems were studied at the Electromagnetic Plant, Oak Ridge, Tennessee. Some chemical problems were studied at Brown University, Purdue University, Johns Hopkins University and by Tennessee Eastman Corporation (TEC) at Rochester, New York. Electromagnetic separation of isotopes is based on the principle that an ion describes a curved path as it passes through a magnetic field. With a magnetic field of constant strength the heavier ions will describe curves of longer radii. Isotopes of an element, since they differ in mass, can therefore be collected separately. The principal equipment required is a source of ions, a magnet to supply the field, a collector for the separated ions, and an evacuated tank to contain the source, collector, and ion path. Much auxiliary and control equipment is also required.

2. The Radiation Laboratory at the University of California was founded by Dr. E. O. Lawrence and his associates in 1936 for conducting nuclear physics research. Discoveries made in Europe were verified by Dr. Lawrence and others and further studied in the 37-inch and 60-inch

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cyclotrons invented by him. Research there proved that only certain uranium atoms exhibited fission properties and indicated that studies of this nature could be accelerated if these atoms could be isolated. U-235, one of three uranium isotopes, undergoes fission with release of tremendous amounts of energy. This phenomenon was the subject of intensive study by physicists all over the world. In 1941 Lawrence's knowledge of U-235's military potentialities resulted in his appointment to head a committee reporting on the subject to the National Defense Research Council. Meanwhile, Dr. A. O. Nier at the University of Minnesota succeeded in separating minute quantities of U-235 with a mass spectrograph of his design, which differed from an earlier Dempster design in deflecting the ions 60° instead of 180°. Beset with theoretical limitations, Dr. Lawrence nonetheless strove to convince the National Defense Research Council that an electromagnetic separation process would work. With UCRL funds the 37-inch cyclotron was converted to a mass spectrograph and proved the design by separating a small sample of U-235 with it. A larger cyclotron, the 184-inch, was then also converted to a mass spectrograph. Studies with this huge apparatus from May 1942 to the summer of 1943 conclusively proved the feasibility of electromagnetic separation. Prior to May 1943, when the District officially took over direction of research contracts, chemical studies of interest to the electromagnetic process were being carried on at UCRL, Brown University, and a pilot plant of Tennessee Eastman Corporation at Rochester, New York.

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2. Operating Arrangements. In June 1943, research for the electromagnetic process was chiefly being carried on at UCRL and Brown University under OSRD contracts. The Manhattan District established liaison with these universities. The District contract with UCRL, authorizing research work, reimbursement, certain employee benefits, patent provisions, return of excess overhead, and other details went into effect 1 May 1943. This contract was administered through an area engineer at Berkeley. The Tennessee Eastman contract provides for research, but its principal provisions call for operation of the full-sized separation plant. This contract was signed 7 June 1943, effective 6 January 1943. Brown University's contract was taken over by the District on 16 April 1943. Its provisions are similar to those of UCRL, except that no employees benefit fund is established. Administration of the Brown contract was first the responsibility of Madison Square Area, later of Chicago Area, and finally, in September 1944, of the Operations Officer of Y-12. Purdue and Johns Hopkins Universities began studies of recovery methods for the Y-12 Plant in December 1944 and February 1945 at the request of the Y-12 Operations Officer. The contracts had originally been administered by the Gas Diffusion Plant Operations Officer and are similar to other contracts regarding reimbursement and patent provisions. They do not provide for an employee benefit fund or return of excess overhead. The research facilities in use by the Electromagnetic Project on 1 May 1943, when the District took over administration of the contract, included ten different Universities of California buildings, in addition to those in the 184-inch area. All new construction was done in the 184-inch area and has included several buildings of temporary construction.

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Facilities at Brown, Purdue, and Johns Hopkins were existing laboratories in the respective chemistry buildings, which had been remodeled where necessary.

Costs of the research work have totalled about $19,500,000, of which over 90% was spent at UCRL.

Security precautions at research stations were those usual for the District and included a continuous guard system, classification of communications, a pass system for employees, and all other procedures to insure utmost secrecy. Safe operating practice and maintenance of satisfactory accident records at research installations have been the responsibility of the District Safety Office and the Contracting Officer.

3. Physical Research. Physical research, dealing with the design and operation of the mass spectrograph for separation of U-235 from U-238 was performed principally at UCRL. The Alpha I prototype was producing material enriched to 9.0% U-235 by 1 May 1943. This equipment was used for training Tennessee Eastman Corporation operating personnel. Improvement programs had the Alpha I unit operating at a 25% higher rate by January, 1944. Beta spectrograph design was studied also. Some of these Beta studies were continued at the Y-12 site under direction of a UCRL staff after 1 May 1943. The Alpha II mass spectrograph, an improved Alpha unit designed at UCRL, was tested at Y-12 in the fall of 1943. Additional work on this unit at UCRL had increased its production rate 50% by October 1944. A "conversion program" to increase production in both Alpha I and Alpha II was studied in 1944. The program is still in progress, and successful units have been operated. An Alpha II program, to increase production by increasing sources and collectors to 18 per units, was terminated because

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of the success of the Gas Diffusion Plant. Some fundamental research has been carried out at UCRL to develop new ion sources and new methods of isotope separation. Basic designs for magnet and tank equipment to be used in Alpha I, and Alpha II, and Beta operations were all drawn up prior to 1 May 1943. Ingenious methods were used to prove this design with small model magnets. Magnetic "shims" to correct magnetic field irregularities were designed and tested at UCRL.

Seventy-two different sources were designed and built at UCRL of "hot" or "cold" types. Source design is complicated by material failures at the temperatures and voltages required. The insulator problem has been especially troublesome. Collectors had to be designed to collect the largest possible fraction of U-235 and either exclude or collect separately the unwanted isotopes. Their design has also been complicated by material failure at the temperatures attained. Graphite, in many intricate shapes, is used in the collector to solve some of the problems. New electrical equipment for control of the mass spectrograph, including high voltage rectifiers, experimental electronic devices, high precision power regulator systems, and controls for high-voltage transient phenomena, was designed at UCRL. Rectifiers supply power considerably in excess of the power used in radio broadcast or other similar high-voltage applications. Power regulators control voltages to 0.1% of average. An extremely high vacuum is necessary inside the mass spectrograph for operation. Diffusion pumps backed by mechanical pumps were developed by manufacturers under the direction of UCRL. These pumps have greater capacity and operate at lower pressures than any other commercial-type pumps.

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4. Chemical Research. Chemical research is concerned with preparation of charge material, recovery of uranium from wash solutions and miscellaneous solids, recovery of enriched material, preparation of final product, and safety hazards involved in each of these operations. Initial research in this field was carried out by UCRL. The Tennessee Eastman Corporation and Brown University made significant contributions. In 1945 Johns Hopkins and Purdue University assisted. Although there was considerable information available concerning the chemistry of uranium by 1 May 1943, there were still many problems to be solved.

Preparation of charge material has been a problem since the start of the project. UCRL and Brown University had developed fairly satisfactory preparation methods by June 1943. The method used produces phosgene, and as a result of one death in 1945 the chlorination method was restudied, not only by Brown University and UCRL, but also by Tennessee Eastman Corporation and Purdue University, resulting in a liquid phase method that had some advantages over the previous method. Fundamental research on the properties of uranium halides has been carried on at UCRL.

The problem of recovering the 90% of UCl_4 charge material, which is normally spattered inside the mass spectrograph, has been one of the principal chemical studies. In Alpha the recovery of enriched material from the receivers was aided by copperplating the receivers at Y-12. The recovery of the enriched uranium from Beta mass spectrographs was facilitated by the use of radioactive uranium. Most of the Beta recovery studies were continued at Y-12 after December 1943.

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Efficient separation of uranium from the wash solutions used in cleaning the mass spectrographs has been a considerable problem. Tennessee Eastman Corporation designed the Alpha recovery system and later the Beta recovery system. Methods of reducing uranium content of wash solutions have been studied by Brown and Johns Hopkins Universities. All solids which become contaminated with uranium must be processed to recover the metal. The graphite used in the collectors sometimes contains 2% uranium, and recovery has been difficult. This problem has been studied by Tennessee Eastman Corporation, Johns Hopkins, and Purdue, but an entirely satisfactory answer has not been found. The highly enriched uranium recovered from Beta receivers must be reprocessed to UF_4 for shipment. Most of the research on this reprocessing has been done by Tennessee Eastman Corporation. Analytical research (studies to determine how much uranium is present in a mixture of elements) and assay research (studies to determine the ratio of uranium isotopes) have been carried out for Y-12 at UCRL and in the Tennessee Eastman Corporation Research Division. Methods for rapid analysis have been developed which give good results in 20 minutes. Assay analysis, following generally the methods devised by A. O. Nier, has been developed to a high accuracy by Tennessee Eastman Corporation. Enriched feed material from the Gas Diffusion Plants has been converted to charge material successfully after a Tennessee Eastman Corporation pilot plant study.

Safety hazard research by UCRL and TEC has been directed at reducing danger of phosgene poisoning, X-ray injury, and poisoning from uranium dust.

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5. Organization. The initial flexible District organization in June 1942 was gradually stiffened until direct contracts between the District and research contractors were effected in May 1943. Key personnel included Colonel J. C. Marshall, Colonel K. D. Nichols, Major W. E. Kelley, Lt. Col. J. R. Ruhoff, Major H. A. Fidler, Dr. C. E. Winters, and Captain J. D. Ireland.

On 1 May 1943 the personnel at UCRL included several of the top ranking scientists in the country and technical people in many varied fields, under the direction of Dr. E. O. Lawrence. UCRL assisted TEC at Berkeley by training numbers of mass spectrograph operators, and later transferred more than fifty key specialists to the Y-12 site.

Tennessee Eastman Corporation did its first research in a Rochester laboratory of the Eastman Kodak Company. TEC research at Y-12 was carried on by the Chemical Development Section until October 1944, and by a separate Research Division since that time.

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MANHATTAN DISTRICT HISTORY

BOOK V - ELECTROMAGNETIC PROJECT

VOLUME 2 - RESEARCH

SECTION 1 - INTRODUCTION

1-1. Purpose. - The initial purpose of the Electromagnetic Research Program was to obtain in the shortest possible time the basic information necessary to design and operate a full scale plant for separation of uranium 235 (U-235) from normal uranium. Research has continued beyond this original objective to increase production through higher yields and lower losses, and to reduce personnel hazards.

1-2. Scope. - The scope of research for the Electromagnetic Project included the electrophysical problems of originating, testing and correcting mass spectrograph design for plant use, and the chemical problems of preparing and recovering uranium charge materials (uranium tetrachloride) and recovering the enriched materials or final product, (uranium tetrafluoride). Most of the physical studies regarding the operation of the mass spectrograph were pursued at the University of California Radiation Laboratory (UCRL). After 1 May 1943 certain of these physical problems were studied at the Electromagnetic Plant, (Y-12), Oak Ridge, Tennessee. Chemical problems were studied at Brown University, Providence, Rhode Island, the Tennessee Eastman Corporation Pilot Plant at Rochester, New York, and the University of California at Berkeley and Davis, California.

The scope of research has changed since October 1943, to include problems concerned chiefly with increasing production in the mass spectrograph, reducing losses in the chemical recovery processes and eliminating

or reducing personnel hazards. Physical and chemical problems with this enlarged scope have continued to be carried out by the UCRL group until the present date. Johns Hopkins University at Baltimore, Maryland, and Purdue University at Lafayette, Indiana, have also been studying some of the chemical recovery problems of the Electromagnetic Plant (Y-12).

In addition to these outside groups, the Tennessee Eastman Corporation has maintained since October 1943, at the Y-12 Plant, a Research Section to study chemical problems and a Process Improvement Division (P.I.) to continue physical research. The activities of P.I. are described in Volume 6, Operations.

1-3. Authorization -

a. All action in connection with the institution and prosecution of this project was taken under authority granted by Congress in the Acts which are described in another book (Book I); the funds used were likewise appropriated by Acts there described.

b. Under the authority vested in him by these Acts, the President issued orders and authorizations which are described in the same book (Book I).

c. Major General L. R. Groves directed or authorized the general policies and directives under which the Manhattan District carried out the work. The S-1 Committee of the OSRD and the Military Policy Committee registered their general approval of the basic decisions involved, as recorded in the minutes of meetings or in other documents in the project files. (Appendix D1; See also Section 5, Organization and Personnel).

1-4. The Electromagnetic Method of Isotope Separation.

a. Process. - This method of separation called for a large number of "complicated electromagnetic devices requiring high vacuum, high voltages, and intense magnetic fields; and a great deal of research and development work." The feasibility of this grew out of earlier work by E. J. Dempster, who in 1918 first used a simplified type of mass spectrograph, and the later work of Dr. E. O. Lawrence who by 6 December 1941, had been able to deposit 1 microgram per hour of U-235 from which a large proportion of U-238 had been removed. The principle of the mass spectrograph is indicated by B1, Fig. 1, Appendix B, this Volume. Here we have the ions of an element with same charge but different masses (235 and 238) accelerated into a vacuum chamber and through a magnetic field. The accelerated ions have approximately the same kinetic energy hence slightly different velocities, (HE $\pm \frac{1}{2}$ Mvg) and different momenta (Mv). This would cause the ions of U-235 and U-238 to follow different circular arcs with the heavier ions having somewhat longer radii than those of the lighter ions. The ions will be most widely separated after completing a semicircle. Since there is only 1.3% difference in mass between the 235 and 238 isotopes* of uranium the problem of obtaining a complete separation of these components is complex and difficult. For example, if the paths in the magnetic field have a radius of approximately 4 feet, the maximum separation of 235 and 238 atoms will be about six-tenths of an inch. Unfortunately, for several reasons, all ions do not hold to their true paths; therefore, there is an overlapping of the paths at the point of collection which causes a varying amount of contamination of one isotope with the other (See App. B1). The diagram "Electromagnetic

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Method of Separating the Components of Uranium (See App. B1), shows a graphic demonstration of the process and the overlapping of ion paths.

b. Equipment Required. - In general, the equipment needed to carry out the electromagnetic method of separation consists of (1) a source for the production and acceleration of ions, (2) a collector for receiving the desired isotope of uranium, (3) a large magnet to make the atoms follow curved paths and (4) a chamber or tank, pumped down to low pressure, in which the particles travel from source to collector. This tank is placed between the pole faces of the magnet. Ions to be separated are produced in the source by an electric arc running in the vapor of a uranium compound. The compound must be vaporized by the application of heat. When the ions of an isotope have been produced they are accelerated by a high voltage and then travel at a constant speed along curved paths in the magnetic field. Upon arrival at the collector the ions give up their charges, are neutralized and solid material is deposited. A high vacuum must be continuously maintained in the tank in which the ions travel, in order to reduce the number of gas molecules present, since when an ion, traveling between sources and collector, collides with a gas molecule, it is deflected from its path. This scattering of ions results in less material collected and poorer separation of the material. Much auxiliary equipment is needed, including vacuum pumps, high voltages supplies, electronic control equipment, low temperature cooling systems, special handling equipment, etc.

A more detailed consideration of the extent of research and development of the various components will be found in Section 3.

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1-5. Historical Background.

a. Establishment of the Radiation Laboratory. - Between 1930 and 1940 Professor E. O. Lawrence and a group of associates in the Physics Department at the University of California were conducting intensive research into the fundamentals of nuclear physics. The importance of these investigations and Lawrence's invention of the cyclotron and its development as a tool of research brought about the establishment of the Radiation Laboratory at the University of California under his direction in 1936.

b. Significant Discoveries.- Discoveries were made during this decade in European and American laboratories regarding the nature and reaction of nuclear phenomena. Discoveries of nuclear reactions of the element uranium under special conditions were promptly verified by Lawrence and his co-workers. Further research into the observed phenomena was carried on at the Radiation Laboratory with such powerful instruments as the 37 inch and the 60 inch cyclotrons. In the course of research both there and in other laboratories, three things became increasingly evident: (1) that only a certain kind of uranium atoms exhibited the unusual properties under investigation; (2) that investigation of these phenomena would be accelerated if a quantity of this kind of atom could be isolated; and (3) that the cyclotrons were ideal tools, readily at hand, that could be adapted for an electromagnetic method of separation of isotopes.

c. Potentialities of Uranium 235. - Uranium has three important isotopes. As it occurs in nature it is composed principally of atoms belonging to the isotope of mass 238; that is, its mass by definition is 238 times the mass of the hydrogen atom. Approximately one atom of uranium in every 140 has a mass of 235; and one atom in approximately 17,000 has

a mass of 234. A characteristic of the 235 isotope of uranium (U-235) is that, under certain conditions, it undergoes a transformation(nuclear fission) accompanied by the release of tremendous amounts of energy. This energy is several million times as great as could be obtained by burning an equal weight of coal. This fission phenomenon, together with its proper control, has been the subject of intensive research by physicists throughout the world. Dr. Lawrence was one of the foremost scientists who attained an early realization of the vast potentialities of U-235 as a military weapon, and he was one of those who took part, in 1941, in the discussions of this aspect of uranium with Dr. Vannevar Bush, Director of the Office of Scientific Research and Development. Dr. Lawrence was appointed to a new committee formed at the request of Dr. Bush, by the National Academy of Sciences to report to the National Defense Research Council (NDRC) of the Office of Scientific Research and Development (OSRD) on this war-vital subject.

d. Separation of U-235 with Mass Spectrometer and Converted Cyclotron.

(1) Early Work. - During 1940, Dr. Alfred O. Nier, at the University of Minnesota, and Dr. K. H. Kingdon, with Dr. H. G. Pollock at the General Electric Research Laboratory in Schenectady, New York, had succeeded in separating minute quantities of U-235 from the other isotopes of the element. Although their process was an electromagnetic method, employing an apparatus known as a mass spectrometer, the rate of separation with their equipment was such that to produce one pound of the material, would have taken some 800,000 years, working 24 hours per day. This calculation was based on the separation and collection of one microgram of U-235 in 16 hours.

(2) Difficulties. - Certain theoretical difficulties, notably space charge limitation, as well as the demonstration of Nier, Kingdon, and Pollock, made the NDRC hesitate to approve the electromagnetic method of isotope separation as a practical approach to the production problem. Lawrence, however, was not convinced that the indicated limitations were insurmountable. In November 1941, he drew on Radiation Laboratory funds and a grant from the Research Corporation of New York to meet the expenses of converting the 37-inch cyclotron to a mass spectrometer, with the hope and expectation of separating a usable sample of U-235. These expenses were later repaid. Within three months the Radiation Laboratory had produced a sample of 100 micrograms. This was a tiny sample but sufficient to be useful in determining the properties of the material and to demonstrate that the method held possibilities of ultimate success.

At about the same time that the 37-inch cyclotron was converted into a mass spectrometer, it was decided to convert the 184-inch cyclotron, then under construction, into a mass spectrometer. The completion of the 184-inch cyclotron, therefore, was indefinitely postponed. Funds to hasten the completion of the huge magnet of this cyclotron were made available by the Rockefeller Foundation as a contribution to the Laboratory's war research.

During the course of this preliminary work the Research Corporation of New York made a grant of $5,000 to the Radiation Laboratory to assist in the isotope separation program. This was in addition to the private funds that had been supplied by the Rockefeller Foundation, the John and Mary Markel Foundation and the University of California. Then, in December 1941, the OSRD offered a contract to underwrite this phase of the research,

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and the grant from the Research Corporation was returned. (For further discussion of contracts see Section (2).

e. Electromagnetic Separation Proved. - After the preparation of the first sample, experiments were pushed day and night to increase the output of the equipment. By March 1942, alterations in the original apparatus had raised the production rate, for short periods, by a factor of 500.

On 26 May 1942, the great 184-inch magnet was turned on for the first time. The tremendous increase in power and flexibilities that this new apparatus (See App. A2) provided the research group was reflected during the following months by corresponding increases in production. By mid-summer 1943, it was demonstrably evident that the electromagnetic method of separating the isotopes of uranium was a practical process and that a large enough electromagnetic plant could have a critical bearing on the war and inestimable implications for nuclear research in the future.

f. Chemistry.

(1) Properties of Uranium. - The element uranium was identified by Klaproth in 1789 in the form of a lower oxide, and in 1842 the metal itself was produced by Peligot (See App. B2). In significant concentrations it is not a common element, but is found in a number of minerals, most of which are rare. The chemistry of uranium has been of interest prior to 1940 principally because uranium ores are a source of radium. The chemical properties of the element were well known before the inception of the electromagnetic project (See App. B2).

(2) Chemical Research at Berkeley. - Prior to August 1942, the only chemistry involved in the electromagnetic process was that necessary for the preparation of the uranium tetrachloride (UCl_4) and other uranium

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halides used as charge material in the mass spectrograph, and for improvement of analytical methods to determine uranium. At this time the work was being carried out at Berkeley, California. About August 1942, a formal Chemical Group for this Project was organized at Berkeley. This group submitted its first monthly Report September 1942 (See App. G1). Besides the problems of preparing charge material, this group also began to consider the problems of recovery of unused charge material and further development of analytical methods.

(3) Research at Brown University. - In September 1942, the Brown University research staff, under Office of Scientific Research and Development Contract OEMsr-290, began studying methods of preparing uranium metal (See App. G2). As a preliminary to the reduction process it became necessary to develop a method for preparing uranium halides. Growing out of this halide investigation UCl_4 was produced and by chance a method devised that became of interest to the Electromagnetic process for production of UCl_4 charge material through the use of carbon tetrachloride in a liquid phase reactor. From September 1943, until 1 June 1943, Brown University (See App. G2, F2) sent 262 pounds of charge material to UCMC for use in the spectrograph.

(4) Status, May 1943. - By 1 May 1943, the date the Manhattan District's contract with UCMC became effective, there existed several processes for making charge material (See App. B3). Also, by this time a small pilot plant was in operation at Berkeley to study methods for 100% recovery of the uranium scattered inside the mass spectrograph at the end of a run. The Tennessee Eastman Corporation contract (See App. D4) was effective as of 6 January 1943, and by February 1943, a few representatives were at Berkeley observing operations there. Some research

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studies were started in Rochester about 15 April 1943. (The first report from this group is in Appendix D5).

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SECTION 2 - OPERATING ARRANGEMENTS

2-1. Coordination of Existing Programs. - In June 1942, research in the fields pertinent to the electromagnetic process was chiefly being carried on at the University of California. This research was authorized by Office of Scientific Research and Development (OSRD) Contracts OEMsr-667,799 and 806. Brown University working under Contracts OEMsr-290 and 688 on Electrolytic process and the preparation of metallic uranium respectively also made contributions through supplying methods and chemical processes for producing charge material. All research on the separation of U-235 had progressed to such a point under OSRD that the War Department was instructed by the President, at this time, to direct the work toward developing a powerful weapon from this material. An Army organization, later designated the Manhattan District, was then established to coordinate and supervise all work (See Vol. 1). In June and July 1942, the new organization established liaison with the University of California and Brown University. An Area Office of the District was placed at UCRL in August 1942. To provide better coordination of the entire project, all of the research development work was transferred to the Manhattan District about 1 May 1943, and new contracts were then prepared for each university to include this change of control from OSRD to the War Department.

2-2. University of California Contract.

a. Negotiations with the University of California. - A letter contract, dated 1 April 1943, was prepared by the District and approved by the University of California Regents as a temporary measure to permit the work to go on while the formal contract was being negotiated

(See App. C3). Negotiations held in the District Offices in New York, on 16 April 1943, resulted in submission of a draft of the formal contract. This contract was entered into by the Government and the University on 20 April 1943, and went into effect 1 May 1943. However, as stated above, the District exercised control over the research at the University of California as early as 1942, through its Area Office. When the formal contract (W-7405-eng-48) was prepared, OSRD contracts OEMsr-687, OEMsr-799 and OEMsr-803 were terminated and the unexpended government property was transferred to the War Department. The research and property thus came under the control of the District. This transfer later included OEMsr-651 when it terminated with OSRD in June 1943.

This contract (W-7405-eng-48) was negotiated with the University of California because of the pre-eminence of the work done there in the field of nuclear physics, the unique equipment, such as the cyclotron, available in the Radiation Laboratory, and the Laboratory staff's extensive knowledge of the separation technique. The research project was in full operation when the Manhattan District formally entered the picture as contracting agency.

b. Provisions of the Contract (See App. D7). - The University of California was authorized to procure the facilities and personnel to obtain the basic information needed to design and operate a full-scale plant for separation of U-235 from normal uranium. The scope of the work included testing and correcting mass spectrograph design, pilot plant studies of chemical charge material preparation and recovery, and problems concerned with removing all health hazards. The scope also included training of personnel for plant operation. All work performed had the prior approval of the Contracting Officer.

The total amount the contractor may be reimbursed is limited by Art. IX, Par. 2 of the contract. Supplement No. 5 to the contract eliminates possibility of a profit to the contractor from the 25% of wages and salaries which he is allowed for overhead.

All employees and their dependents are indemnified for injury due to the hazards of the work by a $1,500,000 fund which will be maintained for a period of ten years after contract termination.

The contract contains the usual patent provisions which give the Government the right to take title to all patents which may result from discoveries made in the course of the work (See Book I Vol. 13 - Patents).

c. Administration of the Contract. - The contractor is assisted in obtaining priorities for material, deferments for key men, and in other details, by the Area Office at the Radiation Laboratory. The Area Office also helps coordinate research at California with research at other sites, and acts as the Government's representative for interpretation of the contract. This Area Office is directly under the Electromagnetic (Y-12) Operations Office at Oak Ridge, which in turn reports to the District Engineer.

2.3. Tennessee Eastman Corporation Contract.

a. Negotiations - The Tennessee Eastman Corporation was first approached concerning the Electromagnetic Project on 24 December 1942. On 6 January 1943, a Letter of Intent was issued and a formal contract was subsequently negotiated on 7 June 1943 (See App. D8). While the principal assignment of the Tennessee Eastman Corporation was to operate the full-sized isotope separation plant, it was recognized by

Title III of the Contract (W-7401-eng-23) that Tennessee Eastman Corporation would have to perform some independent research work to insure production. The research work was subject to the written approval of the Contracting Officer.

b. Provisions of the Contract. - (See Vol. 6 and App. D9). This was not primarily a research contract, but provided for operation of the plant as described in detail in Volume 6. The research work provided for under Title III was to be reimbursed and development (including Process Improvement) was estimated at $1,226,625 for the first year, June 1943 to June 1944.

c. Administration of the Contract. - The Tennessee Eastman Corporation at Oak Ridge is assisted in all phases of its work by the Y-12 Operations Officer, who fulfills the function of an Area Engineer. The Y-12 Operations Officer has liaison personnel on his staff who maintain close contact with both the chemical and the physical research conducted at the Y-12 Plant.

2.4. Brown University Contract.

a. Negotiations with Brown University. - A small research group at Brown University was active in certain branches of uranium chemistry from 1 January 1942, for OSRD (See App. C2). With the agreement of Brown University the two OSRD contracts were replaced by War Department Contract No. W-7405-eng-75 on 16 April 1943 (See App. D9[10]). Since that date Brown University has continued to supply information to the Electromagnetic Project in addition to helping other District plants with problems requiring research.

b. Provisions of the Contract (See App. D10). - Brown University was authorized to procure facilities and personnel to study methods of preparing uranium tetrachloride charge material which could be adapted to quantity production, and methods of recovering uranium from dilute solutions. All work performed had prior approval of the Contracting Officer.

The contract contains the usual total reimbursement limitations. Supplement No. 3 eliminates possibility of profit from overhead. Patent provisions in the contract give the Government the right to take title to all patentable discoveries. No benefit fund was established since the work carried out here was not extra-hazardous.

c. Administration of the Contract.-The Area Engineer, Madison Square Area, had charge of the administration of this contract until March of 1944. At that time, since Brown University was doing over half its work for Chicago, the Chicago Area Engineer was made responsible. In September 1944, all the Chicago studies were completed, and Brown's research was again totally for the Electromagnetic Project (Y-12). Since November 1944, the Y-12 Operations Officer has had charge of the technical administration of the contract.

2-5. Johns Hopkins University and Purdue University Contracts.

a. Negotiations with Johns Hopkins University and Purdue University. - In December 1944 production difficulties at the Tennessee Eastman Plant were such that only a little more than 20% of the uranium metal put into the Alpha Building came out as product (See App. D17). The Y-12 Operations Officer indicated a desire to conduct research at Johns Hopkins and Purdue to help solve this critical production difficulty

(See App. C4 and C5). Both universities indicated their willingness to continue research on the Y-12 production problems since they were performing similar work for the Gas Diffusion Project under the administration of Madison Square Area. Accordingly, Supplement No. 10 to the Johns Hopkins Contract W-7401-eng-43 and Supplement No. 5 to the Purdue Contract W-7405-eng-74 were approved in December 1944 so that research work could continue at these two universities. (See App. D13 and D14)

b. Provisions of the Contracts. - Both Purdue and Johns Hopkins were authorized to study methods of recovering uranium from certain process materials (See App. C5 and C6). The scope of work at Purdue has included construction of a small pilot plant to study loss sources, and some studies of new charge material production processes. All work performed has had prior approval of the Contracting Officer (See App. C7 and C8).

Each of these contracts contains the usual total reimbursement limitations. Neither contract at present contains a supplement to eliminate possibility of profit on overhead, nor do they provide for a special employees' benefit fund. Patent provisions in these contracts give the Government the right to take title to all patentable items.

c. Administration of the Contracts. - During December 1944, personnel at Purdue University were shifted to Y-12 problems so that by 1 January 1945 all technical and administrative supervision of this work was under the Y-12 Operations Officer. Personnel at Johns Hopkins University were shifted during December 1944 and January 1945 to Y-12 problems, so that by 15 February 1945 control of research there was also the responsibility of the Y-12 Operations Officer.

2-6. Eastman Kodak Rochester Laboratory. - One laboratory of the Eastman Kodak Corporation at Rochester was rented by the Tennessee Eastman Corporation on TEC Purchase Order No. 6-4315, for pilot plant studies between April and August 1943. These studies included chemical charge material preparation and recovery of uranium from process solutions. This pilot plant was closed in September 1943 when the last of its personnel were transferred to Y-12.

2-7. Facilities at the University of California.

a. Existing at Start of Contract. - On 1 May 1943, the effective date of the contract, most of the activities of the Radiation Laboratory were being carried on in the 8.3-acre area surrounding the 184-inch Cyclotron Building two miles from the University Campus at Berkeley (See App. B2). Research was also being done on the Berkeley campus proper at Davis (The University of California School of Agriculture). Ten different buildings were being used at these two sites.

The Cyclotron Building, center of physics research and testing, has a ground floor area of 21,000 feet (See App. A3 and B2). In addition to the huge 184-inch magnet, it houses the experimental magnet and several special maintenance shops. There are also offices, a conference room, a storeroom and a first-aid room in this building.

The main laboratory machine shop in the 184-inch area was placed in the same building with the project's receiving and stores department (See App. A4). In this building, there was an assembly shop, sheet metal shop, pattern shop, electronics construction shop, and an electronics research laboratory. The total floor area was over 16,000 square feet. A few special tools were installed for fabricating the complex graphite parts required in the mass spectrograph.

A new laboratory and office building to the west of the Cyclotron Building (Lab. Annex) was nearing completion. It was of temporary construction with 16,000 square feet of floor area.

On the main Berkeley campus the Old Radiation Laboratory was being used entirely for electromagnetic research. The 37-inch cyclotron magnet with which Lawrence produced his first significant samples of U-235 was located here. Also located in this Old Radiation Laboratory Building were a machine shop, a glass-blowing shop, a magnetic research laboratory and a chemistry laboratory.

Other buildings taken over for the project on the Berkeley campus were Le Conte Hall (Physics Building), Donner Laboratory (Medical-Physics), Durant Hall (Administration Offices of Government Patent Group, Stone & Webster, Westinghouse, General Electric, and Tennessee Eastman), and Crocker Laboratory (for radiation tests with 60-inch cyclotron).

The project had four additional chemistry laboratories in the new Chemistry Building at Davis.

b. New Facilities Constructed (See Map, App. B2). - All new construction was done in the 184-inch magnet area. These new buildings, of temporary construction, are shown on the map, and include a large machine shop, two laboratories, a warehouse, a pattern shop, carpenter shop, print shop and smaller buildings.

2-8. Facilities at Other Sites.

a. Brown University. - The Brown University work is carried out on one floor of a modern laboratory building known as Metcalf Laboratory. None of the building facilities were constructed by the District.

b. Johns Hopkins University. - The first floor of the east wing of the Chemistry Building (Remsen Hall) and parts of the basement under this wing, have been devoted to the project work. No new facilities were constructed on the campus, although Remsen Hall itself was altered considerably in the area where project work was carried out.

c. Purdue University. - Most of the special instruments in the Purdue Chemistry Building are available for project use, in addition to one medium-sized laboratory, two small graduate laboratories, use of a machine shop, and office space. The Locomotive Laboratory, an old building near the Chemistry Building, has been remodeled for District work. It contains a pilot plant for reproducing certain Tennessee Eastman chemical processes on a small scale.

2-9. Costs of Research. - The costs of research which are available are somewhat arbitrarily chosen. In a plant such as that of Y-12, where there was little opportunity for development work or pilot plant studies before a full-sized industrial plant was designed, it is not possible to state where research ends and standard plant practice begins. Research costs are defined as the costs of all outside research contractors plus the cost of the Research and Development Division of Tennessee Eastman Corporation.

The following list tabulates these costs:

	Total Spent for Y-12	% of Total Research Costs
University of California	$ 18,000,000	91.4%
Tennessee Eastman Corporation	1,300,000	6.6%
Brown University	189,328	1.0%
Purdue University	98,000	0.5%
Johns Hopkins University	61,000	0.3%
	$19,648,328	

(See App. #11 through #18 for charts of these costs broken down into monthly totals. Cost of the Tennessee Eastman Pilot Plant, approximately $108,000, is included in operating costs listed in Vol. 5.)

2-10. Precautions for Security. - In general, the same precautions for security which obtained at outside research organizations existed at Y-12 plant (See Book I, Vol. 14).

Research contractors were required to clear personnel with local security officers before employing them. All visitors to a research area had to have special visitor's passes which had been approved by the Engineer Office responsible for the area.

Each research laboratory maintained a 24-hour guard force to make certain that no security regulations were violated.

All documents describing phases of the work were classified in accordance with War Department Regulations, carefully watched during working hours, and kept under lock and key at night.

Specially trained officers of the Security Section of the Manhattan District kept close watch on the research laboratories and made periodic reports.

2-11. Safety. - The District Safety Office working through Area Engineers and Contracting Officers has been responsible for insuring safe operating practice at all research installations of the District, and for keeping records of all accidents. A discussion of the Research Safety Program will be found in Book I, Volume II of the Manhattan District History.

Many of the safety hazards encountered at UCRL were unique to the electromagnetic method of production. For instance, the powerful magnets such as that pictured in Appendix A2, caused unexpected accidents. A workman near the magnet at the time it was turned on would find himself in danger of being crushed by a heavy push truck which had been left within the area of intense magnetic attraction and was rolling uncontrolled to the magnet itself. Operators had to be careful when they had metal tools in their hands near the magnets. The magnets attract some metal objects with enough force to give a man a painful fall if he is holding tightly to the metal object.

A safety engineer was at first assigned to the Area Engineer's Office at Berkeley, California, to assist in reducing hazards there. The accident incidence there became so low that he was given other assignments.

Brown, Purdue and Johns Hopkins University research areas are inspected periodically by representatives of the District Safety Section.

All research staffs submit monthly safety reports which list injuries and time lost.

SECTION 3 - PHYSICAL RESEARCH

3-1. General Description and History. - Physical Research, the most extensive of all the research work, deals with the design and operation of the mass spectrograph. The function of the mass spectrograph is separation of the desired U-235 isotope from U-238. While most of this research was performed at UCRL, some phases were studied at Y-12. (The flow diagram in Appendix B10 will assist the reader in visualizing the purpose of the studies described in this section).

a. Alpha I Equipment.- By 1 May 1943, material consisting of 9.0% U-235 and 91% U-238 (See App. D15) was being produced in the experimental XA magnets at University of California (See App. A2) with a source and collector unit designed similar to those later used at the Y-12 plant in Alpha I or "first stage" process. The problem of making this source and collector a smoothly operating production unit remained to be solved. Intensive effort was made to remove the mechanical and electrical difficulties (described in detail in par. 3-2 to 3-5) in order to reduce loss of operating time, and to increase purity and quantity of product. At this same time, and until January 1944, personnel were trained in the operation of this XA magnet so that they could operate the Y-12 plant. By January 1944, units of the Alpha I type in the 184-inch Laboratory at California were producing at a 25% higher rate than in May 1943, and the concentration of U-235 produced had increased to about 13% (See App. B1).

b. Beta Equipment. - Consideration had been given, prior to 1 May 1943, to the problems of obtaining almost pure U-235 by means of a second stage (Beta) process which would use enriched Alpha product material converted to UCl_4, as charge (See App. D16). These studies

raised special problems of mechanical and electrical design. Source design problems were carried out at University of California until 1 May 1943. After this date the studies were continued at Y-12 (See App. B1). These studies are more fully described in Volume 6 since they were a function of the operating contractors.

c. Alpha II. - Studies were made of an improved type first stage unit (Alpha II) also prior to 1 May 1943. This unit was designed to obtain more sources (four instead of two) and higher production capacity than the Alpha I units, with comparable concentration of U-235 (See App. A6 and A7). Several preliminary designs of Alpha II units were made, so that actual testing of the plant unit did not begin until the Fall of 1943. An improvement program for these units extended until November 1944. Success of this program is indicated by the fact that, in November 1943, the Alpha II equipment was producing material 7.5% U-235, with considerable lost time because of mechanical and electrical difficulties. Eleven months later, the equipment was producing material of 13% concentration at about 50% greater rate (See App. D1). In October 1944, it became evident that charge material enriched in U-235 by the diffusion process would be available for use in Alpha II plants (See App. D1). A program was begun at Berkeley to design and test special linerse for the spectrograph to prevent loss of this enriched and expensive material. This design was successfully completed by February 1945 (See App. D18).

d. Increased Production. - In June 1944, a program was undertaken at UCRL to increase output of the Alpha I plant by doubling the

the number of ion sources. Shortly afterward, the same program was started in Alpha II. This work is known as the "conversion program", and had for its purpose the increase of from two sources to four sources in Alpha I and the increase of from four sources to eight sources in Alpha II. By June 1945, successful units had been operating, although an improvement program was continuing as of the date of the Alpha shut down, 15 August 1945.

e. Alpha III. - This program was pursued at UCRL to insure adequate production from the Electromagnetic Process even if the Gas Diffusion Plants failed. The highly complicated Alpha III unit, designed with eighteen sources and eighteen collectors, had about five times the production capacity of the Alpha I unit (See App. D19). The Alpha III design was tested and found practical. It was never used because of the success of the Diffusion Plants.

f. Fundamental Research. - Some fundamental research at UCRL concerning development of new ion sources (substitute K's) and the possibility of separating isotopes by a radio frequency might possibly lead to radical improvements in process. It was found that through the change in the form of the K filaments there might be an increase in effective ionization. It was also thought that a special ion source might be devised to take the place of the hot filament in the K possibly along the same principle as the Philips ion gauge, i.e. a cold source, high potential ionization in the J chamber.

A second major problem concerned itself with an induced radio frequency attuned to that of the ions of the U-235, the wave length of which differed from that of U-238.

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3-2. Magnet and Tank Equipment.

a. Model Studies. - Designs for the magnets and process equipment for Alpha I, Alpha II and Beta were drawn up prior to 1 May 1943. In the case of each unit, performance tests were carried out on prototypes or scaled models. Special methods had to be devised for sensitive measurement of magnetic fields so that deviations of the order of 1.1% in homogeneity of the fields could be detected. This measurement was particularly difficult with the small models (See App. B20 and B21). In spite of the serious overheating of the small overloaded magnet coils, which did not permit more than a few seconds continuous operation, the design of the Alpha I and Beta models was proved practical by 1 May 1943 (See App. B1). Tests were also made on a model Alpha II unit (See App. A5). Later than 1 May 1943, another model AlphaII unit underwent tests and gave excellent performance. The final design allows for magnetic field roughly 35% higher than in the early Alpha II design (See App. D22).

b. Shim Equipment. - Magnetic irregularities in the process tank have considerable effect on the production rate of a unit. The Laboratory at California studied these effects, and attempted in a large measure to correct them through use of "magnetic shims" (See App. D23 and D24). "Magnetic shims" are carefully formed sections of iron which are fastened to the sides of a process tank (See App. A5). They are attached in such a way as to change the paths of the ions to increase their useful angular range as they leave the source.

c. Special Magnets. - Three small magnets were constructed so that special problems could be studied in the laboratory without loss of operating time on the main magnets.

3-3. Source Design.

a. General - The aim of "source" research was to develop an ion emitter unit giving as high a production rate as possible which would operate smoothly over a long period. Units with the most promising performance were carefully investigated and refined before being adopted as plant designs. An indication of the complexity of a plant-type unit is shown in Appendix A6, which shows Alpha I and Alpha II units.

b. Extent of Program - Seventy-two different "source" units were designed and tested at California. All sources fell into one of two general classes, "hot" or "positive"; and "cold" or "grounded". The "hot" type is maintained at high positive voltage while the "cold" type is at ground potential. These two types require considerably different auxiliary mechanical and electrical equipment and are not easily interchanged. It is not possible to discuss the characteristics of these various types without including technical details beyond the scope of this volume (See App. D25 and D26).

c. Design Problems. - Troublesome mechanical and electrical difficulties were encountered such as electrical short circuits, warping of parts, melting or weakening of parts by electrical discharge, and loss of power through discharges. In many cases parts had to be redesigned and more suitable materials sought to solve or lessen the difficulties. The design of the source is complicated by the fact that it must dissipate a large amount of power. This power is due to the required heating for the subliming of the charge material (uranium tetrachloride), to the power in the ionizing arc, and to the high voltages needed to speed up the ions produced in the arc region.

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If this power is not properly dissipated, parts warp to uselessness, and the equipment is seriously damaged. Power in electrical discharges is sometimes great enough to melt and sputter metal parts and to crack and fuse insulators. The high voltage insulator problem has been serious. A thorough search has been made to find the best insulation material to withstand these severe conditions. The insulator problem has had especially high priority in the development of positive type sources. Before a unit was finally adopted, it was necessary to simplify it as much as possible so that non-technical operators would be able to operate the unit.

3-4. Collector Design.

a. General. - The aim of the "collector" (which catches the separated material) design has been to produce a unit which collects a large fraction of U-235 ions in one compartment of the collector and the U-238 ions in another compartment. Some consideration also had to be given to designing a collector from which the product could be removed easily. An idea of the amount of development work carried out at Berkeley can be gained by noting that 117 different types were investigated. This number does not indicate the large number of modifications made in the mechanical and electrical equipment before satisfactory collectors were obtained (See App. D1).

b. Collector Types. - There have been two general major classifications of types of collectors studied at Berkeley; the one-pocket and the two-pocket types. The one-pocket collector was designed for use in Alpha I and Alpha II where the impoverished material is not recovered. The two-pocket collector was designed for Beta units, where

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the charge material used is already enriched in U-235. All Beta charge material which does not reach the collector must be recovered from the tank because of its value. The heavy component is almost pure U-238 and it must be collected separately to avoid dilution of the uncollected, enriched material which is recycled. Hence, the Beta collectors have two pockets (See App. D1).

c. Design Problems.- Design of collectors (See App. D27) has meant determining the best possible slot shape in front of the pocket (See App. A7) to receive a maximum amount of U-235 product. Besides this requirement, the collector had to retain material collected. In operation a large amount of power is delivered to the collector by incoming ions (roughly 20 KW for Alpha II). As in the source, this creates the twin problems of warpage and melting of material, and resulting short circuits. To overcome this difficulty required finding and using the most suitable materials. Graphite has been the most widely used material to protect insulators and collector parts to date. Following an extensive series of measurements to ascertain the best slot shape, satisfactory designs have been found for Alpha I and Alpha II and Beta slots. Some research has been carried out on a "sump" type collector. The "sump" is designed to slow down the incoming ions with a retarding voltage and does not require the use of magnetic shims. The "sump" collector cannot yet compete with the conventional type collector because it does not collect a large enough fraction of the desired isotope. Collectors have also been designed which used electric shims instead of magnetic shims (See App. D26).

This program was dropped because of the high material losses and many mechanical difficulties.

3-5. Electrical Control Equipment.

a. General. - The studies and operation of the mass spectrograph required the design of electrical control equipment to meet demands. These design problems fall into four broad classifications: (1) high-voltage, high power rectifier systems with auxiliary control equipment (2) special electronic devices for various experimental programs (3) high-precision regulation of power supply systems (4) control devices for certain types of high-voltage transient phenomena due to the character of the separation process. The electrical equipment necessary for operation of the racetracks includes magnet power supply and control (See App. D29), power supplies for arcs and for heating the charge containers in the source, high voltage rectifiers and regulators for the ion accelerating system and auxiliary metering and control equipment (See App. D30, D31).

b. Design Problems. - The early high-voltage rectifier units were designed to supply currents of about 0.75 amps at voltages approaching 40 kilovolts. Recently designed rectifier units are capable of supplying 6.0 amperes at voltages in excess of 50 kilovolts. These requirements are far above those encountered in radio broadcast and similar high-voltage power applications and have required extensive studies of the various rectifier systems and tubes which might be employed for this service. Special tube types have been developed. It is interesting to note that the maximum permissible variation in the value of the high-voltage applied to the accelerating system

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is 0.4% of the average voltage. Such precise regulation of large amounts of power at high voltages has required much engineering effort. Appendix A8 shows typical high-voltage equipment. The magnet for an Alpha II half track is supplied by two generators driven by a single motor. The direct current, thus produced, is at the rate of 3000 KW, or at 600 volts and 5000 amps. An electrical engineering department was established primarily to produce the electrical circuit and equipment designs necessary to the basic research program at the Radiation Laboratory. This group later acted in an advisory capacity to Stone and Webster Engineering Corporation on the construction of Y-12.

3-6. Vacuum System.

a. General. - Process tanks can be operated only when the pressure within the tank is close to an absolute vacuum (0.00001 millimeters of mercury). This high vacuum is necessary to prevent scattering of the accelerated ions due to collision with gas molecules, and to avoid breakdown of the high voltage. The vacuum must be maintained in spite of the continued generation of gases during the operation of the unit. Large diffusion pumps backed by mechanical pumps are required to produce and maintain the degree of evacuation at which the tanks operate most successfully. No vacuum pumps capable of operating at such high pumping speeds and low pressures were commercially in use in any other process.

b. Pump Investigations. - Comparative tests were run with pumps up to 32 inches in diameter designed by several contractors in 1944 (See App. D32). Since that time Distillation Products Company under Contract W-7407-eng-155, has developed [illegible] performance to any previously designed. [illegible] diffusion pump with

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square cross-sections, for more economic space arrangement are now in progress. Some of the pumping equipment developed at Berkeley is shown in Appendix A9.

c. Instruments. - Apart from the emphasis on development of pumping equipment, research has been directed to developing suitable pressure measuring devices for use in the process tank and connecting lines. A new-type instrument has been invented (Annie) for indicating the individual components of the residual gas (See App. B33).

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SECTION 4 - CHEMICAL RESEARCH

4-1. General Description and History.

a. Scope. - Chemical Research deals with the preparation of charge material for the mass spectrograph, removal of uranium from the spectrograph after a run, recovery of uranium from wash solutions and miscellaneous solids, recovery of enriched material and preparation of final product, analysis of solutions and assay of uranium, and investigations into the safety hazards involved in each of these operations. (For help in understanding the applications of the research problems see Flow Diagram Appendix H10).

b. Research Groups. - As stated earlier (See Sec. 1), the Tennessee Eastman Corporation started a research group at Berkeley in February 1943, and another group at Rochester in April 1943. In May 1943, there were three organizations conducting research on the chemical problems of the Electromagnetic process. These groups were the University of California Radiation Laboratory (UCRL), Tennessee Eastman Corporation and Brown University.

c. Information Available. - There was considerable information available in May 1943, related to the chemical problems of the electromagnetic process. However, there were also numerous problems to be solved before the laboratory scale chemistry of Berkeley could be transferred into the full-scale plant being built at Oak Ridge (See App. D4). The state of the chemical research was advanced far enough to permit Tennessee Eastman Corporation to prepare flow sheets for both Alpha and Beta production (See App. H10 and D34).

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The essential features of the recovery processes used at Y-12 are contained in these flow sheets.

The principal research problem during the middle of 1943, was the production of satisfactory charge material. Two methods, vapor phase (UCRL) and liquid phase (Brown University) existed. Both processes had serious disadvantages for quantity production. The liquid phase process was studied in the Tennessee Eastman Kodak pilot plant at Rochester (See App. D35).

4-2. Preparations of Charge Material.

a. Types. - Several materials were tested before it was decided that uranium tetrachloride was the most satisfactory (See Appendix G1). In December 1942 the compound hexachloride (UCl_6) was discovered at Berkeley. This compound seemed to hold possibilities for use as a charge material but by June 1943 it was no longer being seriously considered because of its lack of uniform vaporization in vacuum and the incident difficulty in maintaining uniform vapor phase pressure from the charge. Other uranium salts such as uranium tribromide (UBr_3), uranium tri-iodide (UI_3) and uranium trichloride (UCl_3), were tested for use as late as September 1943, (See Appendix D37). These compounds were found to be less satisfactory than the tetrachloride because of their greater difficulty in preparation and their lack of stability in process. (See Appendix D37).

b. Tetrachloride Studies. - Many methods of producing uranium tetrachloride existed by September 1942 (See App. G1). However, the problem of producing quantities of UCl_4 as a satisfactory charge material was subject to concentrated studies during 1943 and has been a source of study and research up to the present

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date, 31 December 1946. The vapor phase method, studied extensively at Berkeley, produced only small quantities of UCl_4. The liquid phase method, originated at Brown University, produced ample quantity of UCl_4 per run but generated an extremely toxic gas (Phosgene) under pressure. (See App. C9 - 13-16). Both methods are subject to preparational difficulties (See App. C9). Research continued at Berkeley during 1944, on preparation of UCl_4 to improve upon the production methods which were put into the plant, (See App. D38). Research has also continued by TEC at Y-12 on the liquid phase method, the vapor phase method and, more recently, on the possibility of using hexachlorpropylene in a liquid phase preparation of UCl_4. (See App. D41). This method of preparation shows promise of simplifying the recovery process and eliminating certain hazards peculiar to the preparation of the charge material.

e. Other Studies. - In the early months of 1945, the problem of producing UCl_4 without a toxic gas by-product (because of 1 death in 1945) was studied by Purdue University and the TEC Research Department (See Appendix D39 & 40). In March 1945 the TEC Research Department discovered a liquid phase method which seemed to have some advantages over the then existing methods. This was the hexachloropropylene method (See App. D41). Brief studies were made at Johns Hopkins University during the first half of 1945 to help determine the physical characteristics of UCl_4 made by different methods. (See App. D42). This information is important because some UCl_4, due to wide variation in temperatures required for vaporization, does not work as well as other UCl_4's in the mass spectrograph. This variation was found to be due to small amounts of impurities and, possibly, to difference

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in crystal size. During this same period fundamental research was carried on at Berkeley to determine properties of uranium halides (See App. D43).

4-3. Recovery and Refinement of Uranium Salts from Mass Spectrograph After a Run.

a. General. - In each mass spectrograph, approximately 90% of the UCl_4 material charged does not reach the receiver, but is splattered in varying amounts over the source and other parts of the machine. It was early realized that recovery of this material, especially in the Beta tanks, was an important problem (See App. C8 pp 16 & 17 and Book V, Vol. 3, p. 3.22). This paragraph deals with the methods used to recover these salts from the machine in a solution or solid which would be re-processed to prepare charge material (See App. D44 and D45).

b. Recovery from Alpha Receivers. - In Alpha, the recovery from the receivers was the only recovery problem considered at length, since all the material splattered about the machine was normal uranium, and could be replaced easily. Recovery of the enriched material contained in the Alpha receivers turned out to be more of a problem than was originally expected. The receivers were made of stainless steel. Early recovery studies made on the 37-inch cyclotron at UCRL indicated that all enriched material could be removed by an acid, water and steam spray wash. However, when the Alpha spectrographs started operating late in 1943, there was an unexplained loss of 50% at the receiver. It was suspected that this "loss" was caused by ions penetrating into the stainless steel since this material could not be recovered by a

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simple acid wash of the receiver. The problem was solved by copper plating the receivers since copper dissolved readily in an acid wash and contained the uranium which formerly would have penetrated into the less soluble stainless steel. This copper plate had to be replaced after each washing.

c. Recovery from Beta Tanks. - In Beta, as noted above, it was necessary to recover all of the uranium charged. More elaborate methods of recovery were employed. (See App. C9 pp. 18-19). A novel method of detecting uranium losses inside the Beta machine was developed at UCRL during the middle of 1943. Radioactive uranium 237* (U-237) was prepared by irradiating the usual charge material (See App. D47). The amount of uranium lost in certain parts of the spectrograph could then be readily determined by the radioactivity, with the U^{237} serving as an indicator, of that part in question by the use of a counter. (See App. D48). By December 1943, nearly all recovery studies were being carried on by TEC at the site of Y-12, and this recovery became an operation problem. Research at Y-12, on removal of uranium from the spectrographs, has included a search for protective coatings, to be used at points inside the Beta machine where a high concentration of uranium impinges on the liner of the machine (See App. D49), and use of sodium with the charge material, to combine with all the free chlorine and reduce corrosion of the machine (See App. D41).

4-4. Recovery of Uranium from Solutions.

a. General. - At first it was believed that the material splattered inside the mass spectrograph might be successfully removed dry. (See App. C1). However, trial runs made on the 37-inch

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cyclotron at Berkeley in November 1942, indicated that at least some parts of the equipment should be washed with hot water and/or dilute acids to remove all of the uranium (See App. D30). Processes for the quantitative recovery of uranium from the wash solutions were suggested at this time, and since then have been one of the most exhaustively studied of all the chemical problems. By March 1943, several purification processes for Beta solutions had been suggested, and a pilot plant was installed at Berkeley for studying them (See App. D31). As in the case of methods of cleaning the spectrograph, pilot plant studies regarding recovery of uranium from solutions were concerned chiefly with Beta problems. By July 1943, three Beta recovery processes were being studied in pilot plants, two processes at Berkeley (See App. D36) and one at Rochester (See App. D52). The Alpha recovery process was designed by Tennessee Eastman Corporation (See App. D53).

b. Beta Studies. - A process known as the oxalate method for recovery of uranium and production of charge material for Beta was evolved by UCRL in July 1943 (See App. D54). It was decided, at a meeting held the second week of October 1943, to install facilities for this process and for one known as the peroxide process being considered by TES, since both the oxalate process and the peroxide process apparently gave satisfactory recovery. (See App. D55). Beta Pilot Plant studies at Y-12 began in December 1943 and have continued to the present date, 31 December ~~XX~~, 1946. An operational difficulty in converting the uranium to the uranous form with the electrolytic cell prevented the UCRL or oxalate method from being

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used as the plant recovery system. The peroxide method was initially handicapped by having too much copper interference in machine wash solutions. Due to this difficulty an ether extraction method was used during a short interim while further improvements were being made on the peroxide method which later became known as the Larsen or Gold Peroxide process. This peroxide method has continued to be satisfactorily used until the present date, 31 December ~~3E~~, 1946.

a. Miscellaneous Improvements. - Considerable research work has been done to determine the best methods of reducing the uranium content of dilute solutions from which the bulk of the uranium has been removed by precipitation. Brown University discovered an organic solvent which could be used to extract uranium selectively from solutions containing only a few parts per million (See App. D56). Johns Hopkins University studied the efficacy of co-precipitating uranium with lime to reduce the concentration of uranium in certain effluents (See App. D57). Recovery studies have been made by the Tennessee Eastman Corporation research labs since December 1944. An extraction column with a rotating element inside was developed by UCRL (See App. D58). It is known as a "spinner" column, and it has possibilities of being far more efficient than the standard counter-current flow columns now in use.

4-5. Recovery of Uranium from Solids. - The value of the enriched material is such that utmost attempts are made to recover it from all the miscellaneous solids contaminated with it. These solids include the graphite electrodes and metal parts used in the mass

spectrograph, filter paper and sponges used in the laboratories and buildings, the uniforms and shoes of plant workers, and in some cases, the surface of floors on which material has been spilled.

Most of these recovery studies have been made by the Tennessee Eastman Corporation. Purdue University and Johns Hopkins University have considered the problem of removal of uranium from graphite (See App. B57). This material contains as high as 2% by weight uranium when it is removed from the spectrograph. Recovery of the uranium from the graphite is most difficult because of the complex nature of the graphite. An entirely satisfactory recovery scheme to remove uranium from graphite has not been evolved.

4-6. Recovery and Preparation of Final Product. - The uranium material removed from the receivers in the Beta spectrographs is the most highly enriched uranium obtained at Y-12. This material is extremely valuable. Before it can be shipped it must be reprocessed into the desired form (uranium tetrafluoride).

Most of the research work incident to the preparation of the final product, such as purification and grinding of the UF_4 salt, done expressly for the Y-12 plant, was accomplished by Tennessee Eastman Corporation. Brown University has recently done some research on an improved method of preparing the final salt (See App. D59).

4-7. Analytical and Assay Research.

a. Definitions. - Analytical research refers chiefly to all of the studies made to permit the accurate analysis of samples for the amount of uranium in the presence of any of the other 92 elements officially known. Assay research has been concerned with methods of determining the [redacted]

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uranium in any sample of the metal or compound of the metal.

b. Analysis. - Since study of analytical methods for uranium is not a problem peculiar to the Y-12 plant there has been considerable interchange of "know-how" between Y-12 and other Manhattan plants. For this reason, it is difficult to trace various analytical methods to their original source. There are many methods of determining uranium. Most of these methods, which are highly technical, are listed in the CEW-TEC Manual of Standard Analytical Methods (See App. D68). Recent analytical research has been directed at finding rapid, simple methods of analysis for uranium, and also at finding methods of determining extremely low concentrations of uranium. Analyses employing fluorescence are rapid and relatively simple. They were studied jointly at Tennessee Eastman Corporation (See App. D60) and University of California (See App. D61). Other rapid methods such as colorimetric, spectro-photometric and polargraphic have been studied extensively at Y-12 by Tennessee Eastman Corporation, and have reduced the time for rapid analyses to approximately twenty minutes.

Two additional organic solvents for uranium nitrate were discovered by Brown University in June and July 1944 (See App. D62). One of these, known as "penta ether", has been used as an analytical reagent to replace highly inflammable diethyl ether. This penta ether extracts uranium selectively with few other elements from very dilute solutions.

c. Assay. - Assay research has been carried on concurrently with routine determination of plant samples at Y-12. Research on assay methods has also been carried out at UCRL for the Y-12 project during 1944 and 1945 (See App. A10). The two methods, which are used in assay work, for determining the percentage of isotope 235 are described in

the referenced reports. These methods had been established by A. O. Nier of Minnesota and others before 1 May 1943 (See App. D63).

Assay research has been concerned with improving the accuracy of the determinations, and the speed with which determinations can be made. It is of interest to note that samples to be assayed must first be carefully purified. This purification procedure involves considerable time, and hence has been one of the subjects of study. At present the accuracy of the assay laboratories at Tennessee Eastman Corporation is indicated by the fact that these laboratories are the only ones capable of determining directly with the mass spectrograph the concentration of the scarce uranium 234 isotope.

4-8. Conversion of Enriched Feed Material. - In November 1944, it became necessary to prepare the Y-12 plant for treatment of enriched material from the gas diffusion and thermal diffusion plants. (See Book VI and Book II - Vol. 5). Research studies were made by the Y-12 research group in November 1944 (See App. D64) to see if satisfactory charge material could be made from this enriched feed (uranium hexafluoride). Satisfactory charge material was made. Later, it was apparent that the gas diffusion plant would be able to feed highly enriched material equivalent to Beta feed, and so extended studies, including a small pilot plant, were begun in May 1945, and have continued to the present date (See App. D65). These pilot plant studies have established a satisfactory plant process.

4-9. Medical Research and Safety Hazard Problems. - By April 1943, it was nearly certain that the chemical process used to produce charge material would have poisonous phosgene gas as a by-product.

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Research was begun at UCRL in April 1943 (See App. D66) to devise an automatic instrument which would give the alarm in case toxic concentrations of the gas occurred (about one part per million). The recorder was completed in November 1943, and shipped to Y-12 (See App. D38).

Control tests, on the hazards of uranium dust and danger from X-rays, were made by a representative of UCRL at Y-12 in December 1943. (See App. D38). These tests indicated that neither dust nor X-rays were likely to be injurious during normal operation.

(See Book I, Vol. 7 for more detailed information on operation hazards at Y-12).

SECTION 5 - ORGANIZATION AND PERSONNEL

5-1. District. ~~SECRET~~

a. Organization. - The initial District organization for Y-12 research as set up in July 1942, was very flexible and was organized on a basis of cooperation and liaison between the working groups. This was partially due to the fact that most of the work was being performed under OSRD contracts. Also, the electromagnetic research was under the direction of Dr. E. O. Lawrence who was best able to plan the work which would be of greatest value to the design job. The District Engineer maintained close liaison through officers assigned to each location and, as the research needs were narrowed, the control was tightened, until, in May 1943, direct contracts between the District and the research groups were effected. From that time on, the District Engineer exercised direct control over all work through Area Engineers or representatives of the Y-12 Unit Chief.

b. Key Personnel. - Colonel James C. Marshall, the first District Engineer, established liaison with University of California and was responsible for early integration of California work in the District Plan. Colonel Marshall was assisted by Colonel K. D. Nichols, who later became District Engineer. Colonel Nichols continued to integrate the research work at University of California through the Y-12 Unit Chief, Major W. E. Kelley, and later through Lt. Col. J. R. Ruhoff.

The Y-12 Unit Chief has been represented at the University of California by Major H. A. Fidler, California Area Engineer, and later at Johns Hopkins University, Purdue University and Brown University by Dr. G. B. Winters and Capt. J. D. Ireland.

5-2. University of California.

a. Composition. - The personnel of the Radiation Laboratory, on 1 May 1943, included several of the top-ranking scientists of the country. These were supported by a carefully selected and competent group of theoretical and experimental physicists; chemists; electrical, electronics and mechanical engineers; laboratory and radio technicians; draftsmen; machinists; and various mechanics. Many of the group had worked on the electromagnetic process since its inception (See App. D1). The graphs in Figure 3 (App. B3) show the monthly fluctuations in Radiation Laboratory employees from 1 May 1943, to date. The research group, for the purpose of the chart, includes physicists, chemists, mechanical research engineers, mechanical design engineers, electrical design engineers, laboratory technicians (above junior grade), reassembly coordinators and electronics coordinators. The table on Figure 3 gives a breakdown into six categories of laboratory personnel at approximately six months intervals. With the exception of shop personnel, it will be noted that the trend for all groups follows the graph. Machinists show a continuing increase because of special production work for Y-12, and expansion in 1944 and 1945 of the Alpha I and Alpha II conversion programs (See App. B4).

b. Organization. - The organization has continued to be essentially the same throughout the Manhattan District's administration of the contract (See Organization Chart, App. B5). It may be noted that the Regents of the University of California, who are responsible for the administration of the institution, have placed an unusual degree of confidence in the Director of the Radiation Laboratory, in the Secretary to the Regents, and in the Contracting Officer of the Government. The Regents have accepted the responsibilities and obligations of these

contracts without asking for any disclosure of the subject work.

c. Assistance to Tennessee Eastman Corporation-

(1) Assistance at Berkeley. - Early in 1943, representatives of the Tennessee Eastman Corporation established an office at Berkeley. During the next eight months a considerable number of Tennessee Eastman personnel visited the Laboratory. These people received instruction for various periods in the fundamentals of the electromagnetic process and in operation of the experimental magnet. This group of Tennessee Eastman Corporation employees later had the responsibility of instructing and supervising the large body of technicians who were hired to operate the Y-12 plants.

(2) Transfer of Laboratory Personnel. - During the summer and fall of 1943, the Laboratory approved the transfer of more than fifty key specialists to the Tennessee Eastman Corporation payroll to assist further in the organization of plant activities (See App. D1).

(3) Assistance at Y-12. - Since August 1943, a group of Radiation Laboratory personnel has been maintained at Y-12 to render continued assistance to the operation. These persons have been of considerable value in starting successive racetracks, in improving operating equipment, and in increasing production. The number of UCRL personnel has fluctuated considerably, as shown in Figure 7 (See App. B4).

5-3. Tennessee Eastman Corporation.

a. Early Organization. - The first separate research done by Tennessee Eastman Corporation was carried out at Rochester, New York, in a space set aside at the Eastman Kodak Laboratory (See App. D4). This group started work about 15 April 1943, and had 26 people by June 1943,

when the staff reached its peak. During July 1943, personnel began leaving for the plant site, so that by the last of August 1943, the Rochester group no longer existed. During the month of October 1943, the Chemical Development Laboratory was put under the Chemical Division at Y-12 (See App. D4) where it remained until 24 October 1944 (See App. D67). On this date Chemical Development became a separate Division of Tennessee Eastman Corporation with 129 employees.

b. Later Present Organization. - The number of members of the staff of the Research and Development Division has increased steadily to 261, which was the total payroll on 16 June 1945. It is organized ~~(June 1945)~~ into six groups under the Director of the Division. Each group in turn has a group leader (See App. B6).

5-4. Other Locations. - Organization charts for Brown University, Purdue University and Johns Hopkins University are given in Appendix B7 through B9. These charts give the key personnel at each location. The number of personnel at each of these places is small, and consequently the organization may be readily changed to facilitate attack of problems assigned through the Y-12 Operations Officer.

5-5. Acknowledgements. - The History of Y-12 Research would not be complete without mentioning general organizations, other than those previously mentioned, which have given valuable assistance to Y-12 without formal contract obligations. Certain metallurgical studies were done at Iowa State College, National Bureau of Standards and Massachusetts Institute of Technology. Health hazards of the Y-12 plant have been considered at the University of Rochester. From time to time special

analytical problems of the Y-12 plant have been studied by Dr. H. H. Furman, of Princeton University and Dr. C. J. Reddick, of the National Bureau of Standards.

MANHATTAN DISTRICT HISTORY

BOOK V - ELECTROMAGNETIC PROJECT

VOLUME 2 - RESEARCH

APPENDIX "A"

PHOTOGRAPHS

No.	Description
1	Plate I - The 37 inch Magnet As Adapted for the Electromagnetic Method of Separation
2	Plate II - The 184 inch Magnet Plate III - The 184 Magnet
3	Plate IV - The 184 inch Area - View Looking South - West
4	Plate V - Main Machine Shop Plate VI - Main Machine Shop
5	Plate VII - Alpha II Magnet Model Plate VIII - Installation of Magnetic Shims in Process Tank
6	Plate IX - Alpha I Unit Plate X - Positive $4J$ Source of the Alpha II Type
7	Plate XI - Collector Used With $2-J$ Source Plate XII - Collector Used With $4-J$ Source
8	Plate XIII - High Voltage Equipment Plate XIV - Control Room
9	Plate XV - Vacuum Diffusion Pumps Plate XVI - Vacuum Diffusion Pumps
10	Plate XVII - Chemistry Assay Laboratory (UCRL) Plate XVIII - Change Vaporization Research

A1 **The 37 inch Magnet as Adapted for the E. M. Method of Separation**

This is the equipment in which U-235 was separated in significant quantities; The mass spectrograph, or tank, in the rectangular box lying on its side between the upper and lower pole-pieces of the magnet. In the right foreground is the vacuum diffusion pump. Note the observation windows in the tank.

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A2 The 18¼ Inch Magnet.

The XA Magnet.

Plate II shows the massive yoke and the top circular pole piece of the 18¼ inch magnet. Two tanks may be seen lying horizontally under the pole piece. To the right of the 18¼ inch magnet is the frame of the XA magnet. The 18¼ inch magnet is located under the huge dome shown in App. A3.

Plate III shows the XA or Alpha experimental magnet which has spaces for two tanks. The black, ribbed cylinders in left foreground are vacuum diffusion pumps for the left tank.

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A3 The 184 inch Area --- View Looking Southwest.

The circular building houses the 184 inch magnet and other electrical equipment. In left foreground is a chemical laboratory. In right foreground is the carpenter shop and main machine shop. The cities of Berkeley and Oakland are in the background. For further identification of buildings see map in Appendix B2.

PLATE IV
THE 184" AREA
View Looking South-West

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A4 Main Machine Shop.

The two views of the UERR machine shops show the many machines required to fabricate parts needed in the research work.

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PLATE V

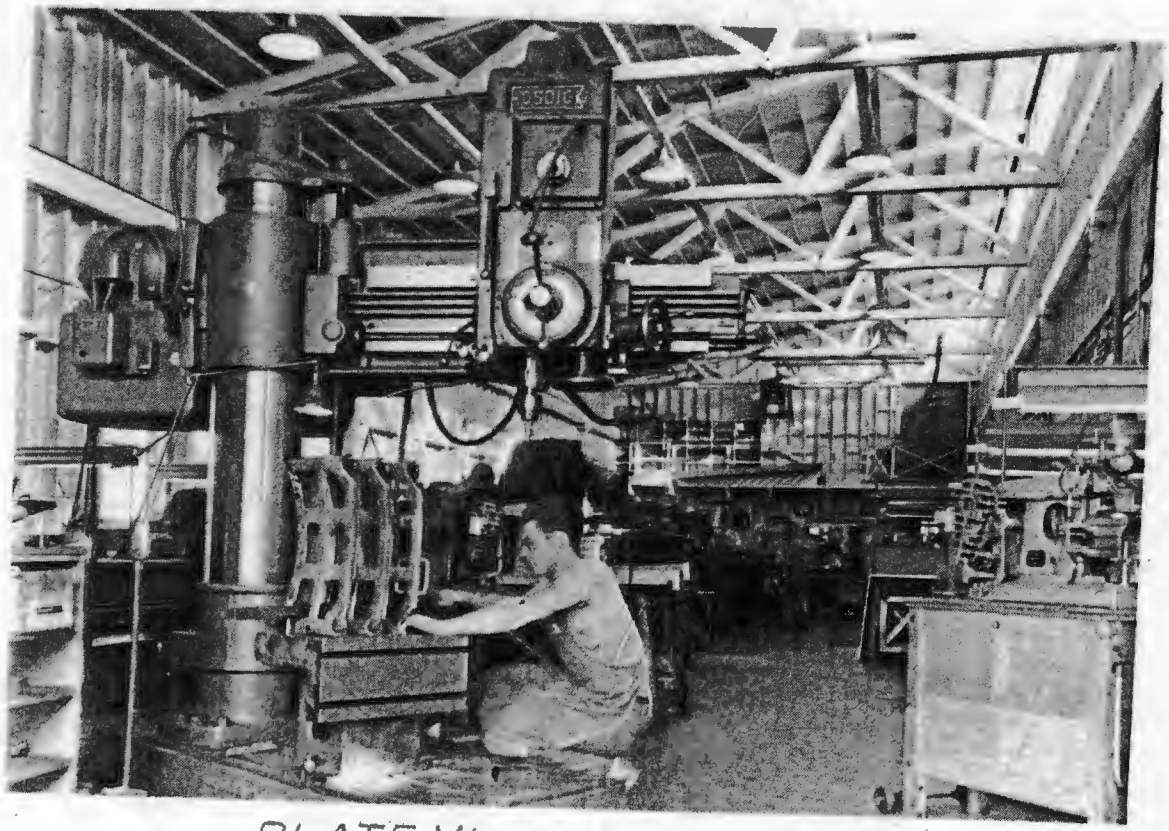

PLATE VI Main Machine Shop

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A5 Alpha II Magnet Model.

Installation of Magnetic Shims in Process Tanks.

A line of small-scale magnet coils is shown in Plate VII. These coils were used to study and check the full-sized plant design of Alpha II.

Plate VIII shows magnetic shims installed in the upper surface of a process tank. The flat metal plates in the foreground are shims ready for installation in the lower surface.

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A6 Side View Alpha I Unit.

Plate IX shows the two sources (right) and the two box collectors (left) in an Alpha I unit. The C-shaped frame above supports a liner which has not yet been installed.

A-7 Source of the Alpha II Type.

The complexity of Alpha II source design may be seen in Plate X. This unit has four ion sources sending four ion beams into a collector similar to that shown in Appendix A7.

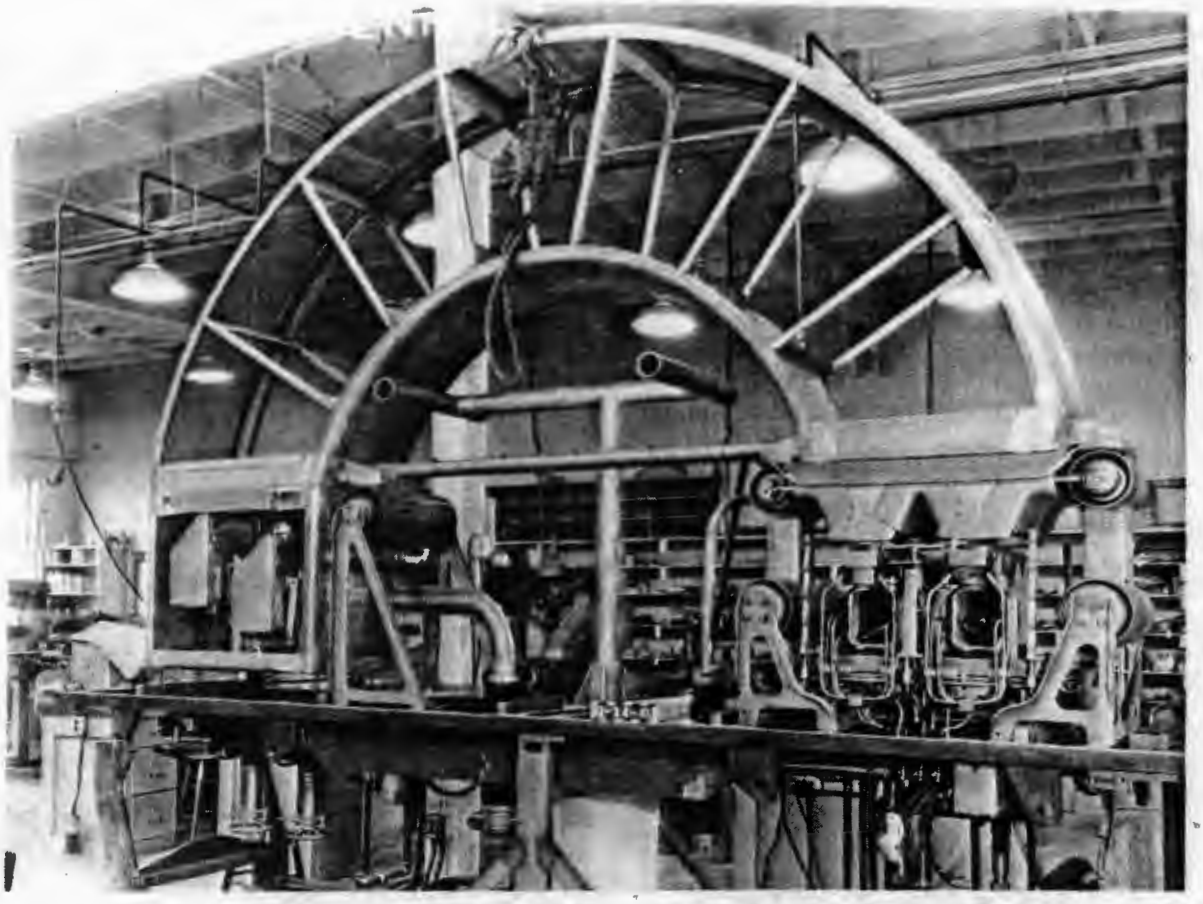

PLATE IX Alpha-I Unit

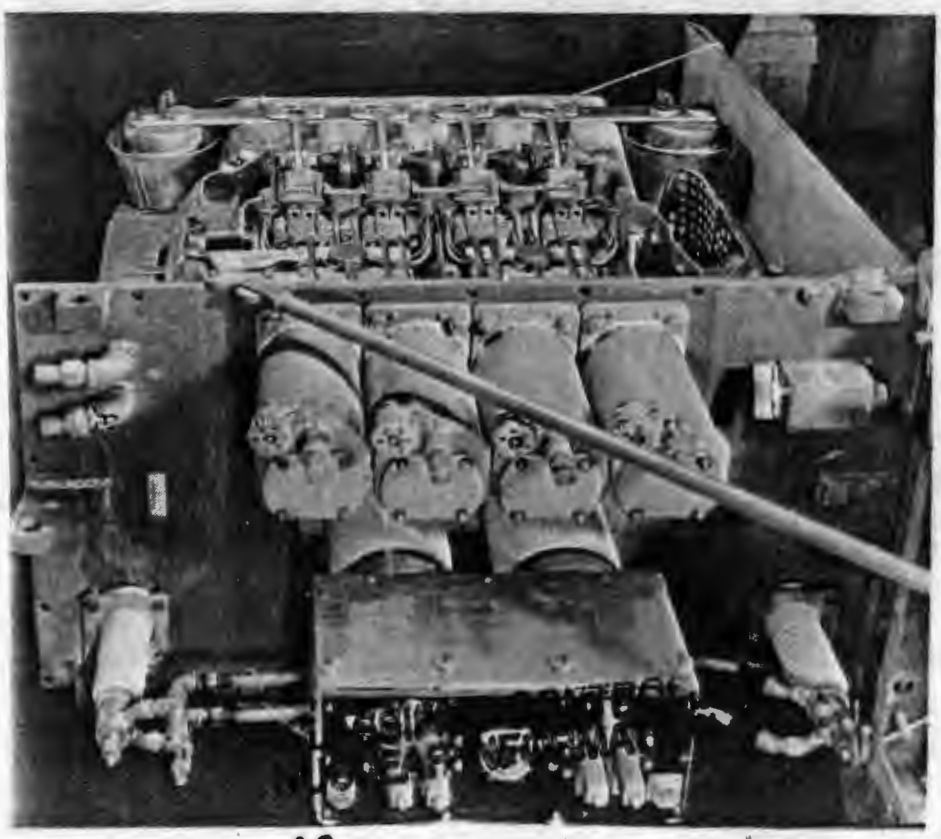

PLATE X
Positive 4J Source of the Alpha-II Type.

A7 Collector Used with 2-J Source.

Collector Used with 4-J Source.

Plate XI is a close up view of two experimental Alpha I collectors similar to those shown in Appendix A6. Note that the left box is open to receive the U-235 isotope, while the right box is closed except for the small slots so that the ion beam can be focussed without filling the box with undesirable U-238. The fronts of these boxes are made of graphite.

Plate XII illustrates the greater complexity of the Alpha II collectors. The accurately-machined graphite fronts of the boxes must be replaced after each run. In this picture, it may be observed that the top box is closed.

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A8 High Voltage Equipment.

Control Room.

Plate XIII shows some of the high voltage equipment used at UCRL which had to be built into a much smaller space at the Y-12 plant. Plate XIV shows the control room used to operate the XA magnet.

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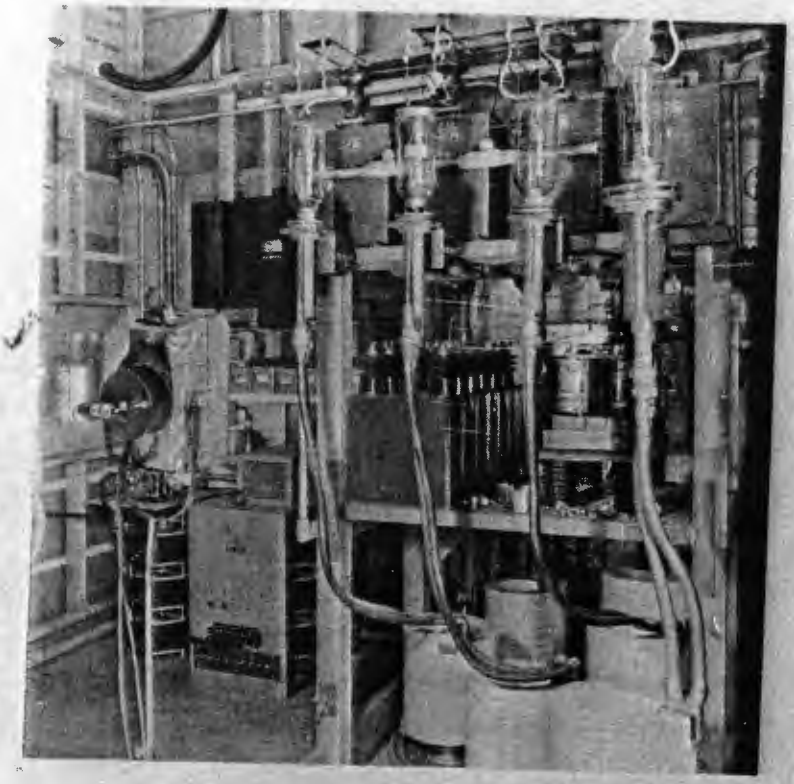

PLATE XIII

High Voltage Equipment

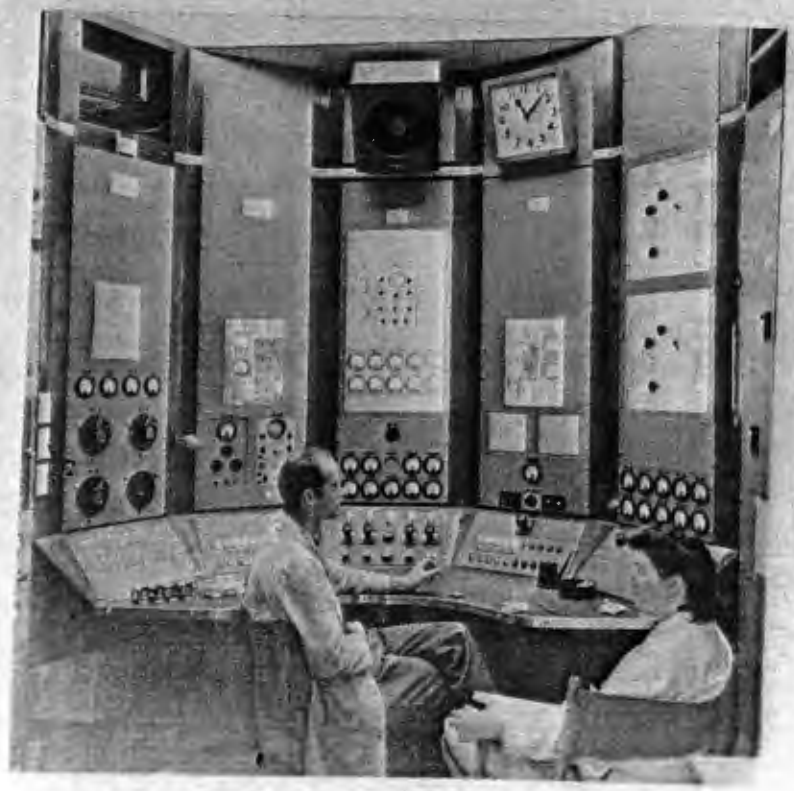

PLATE XIV

Control Room

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A9 Vacuum Diffusion Pumps.

Two views of oil diffusion pumps developed for the electromagnetic process are shown. The large 30-inch pumps shown in Plate XV are the type presently used in Alpha II. The smaller pumps in Plate XVI were used at UCRL with early experimental units.

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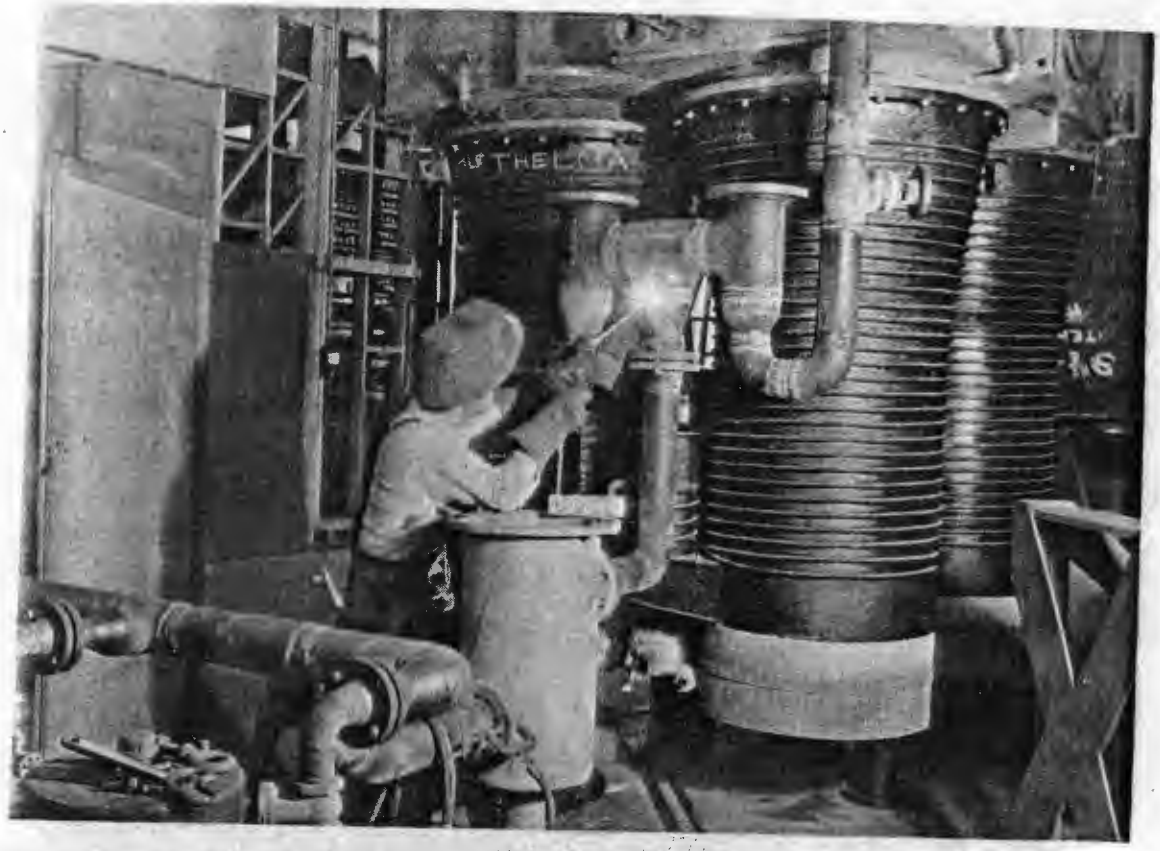

PLATE XV

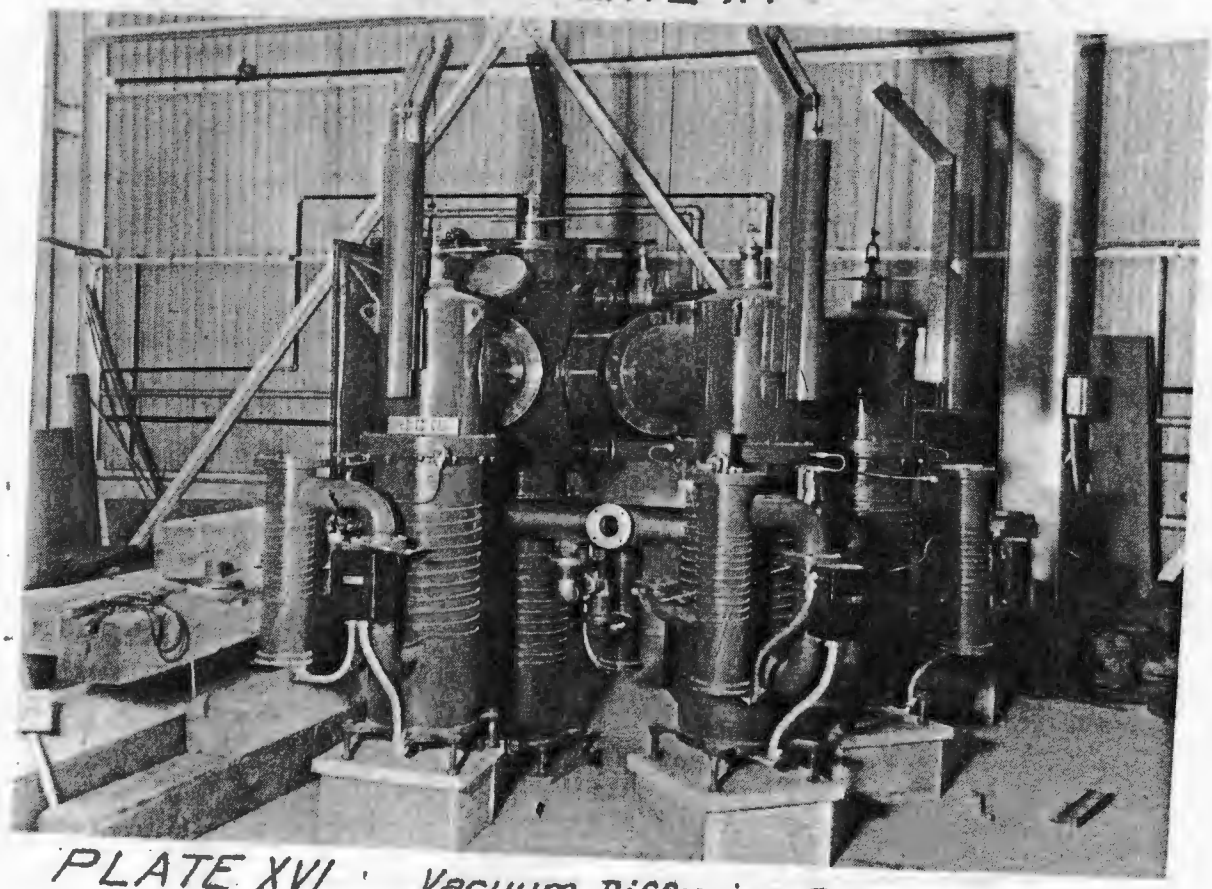

PLATE XVI · Vacuum Diffusion Pumps

A10 Chemical Assay Laboratory.

Charge Vaporization Research.

The assay laboratory at UCM. is pictured in Plate XVII.

The Plate XVIII shows equipment used at UCM. to study the volatilization characteristics of different charge materials.

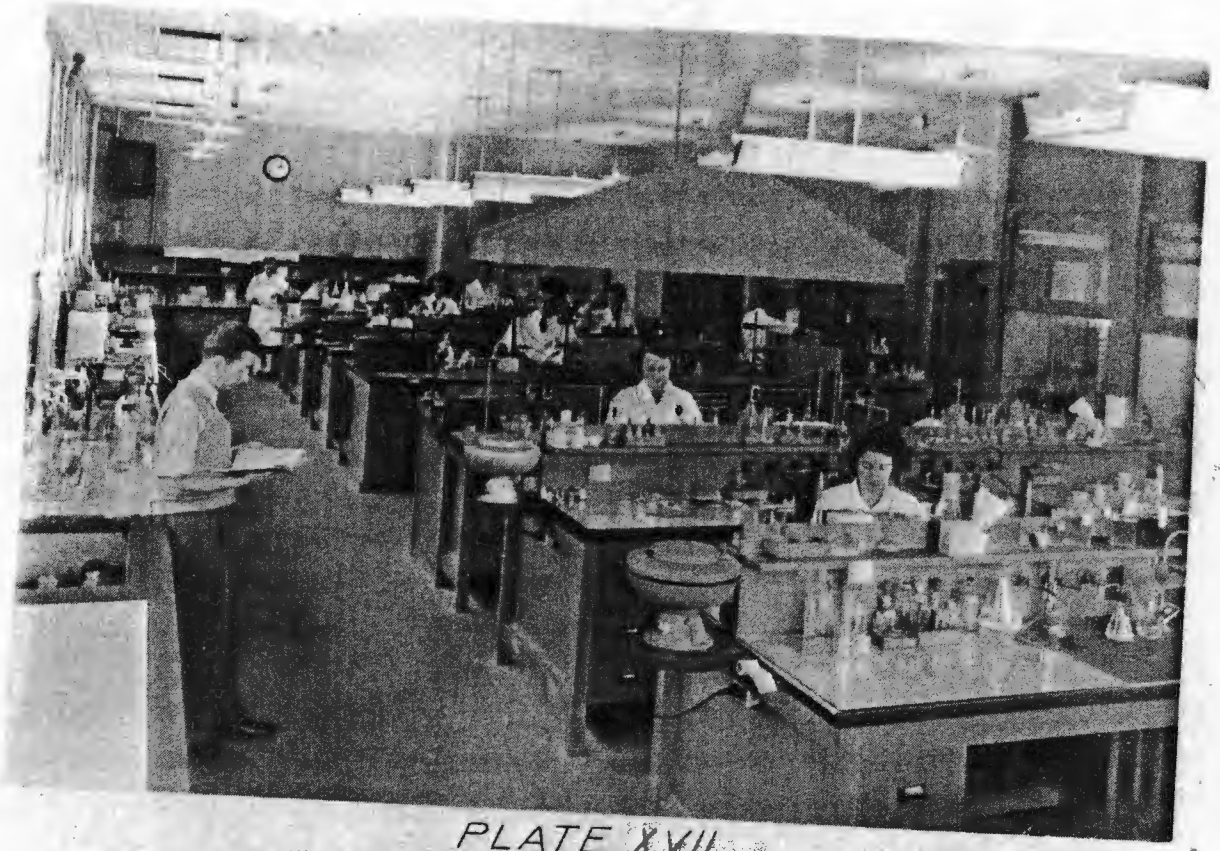

PLATE XVII
Chemistry Assay Laboratory

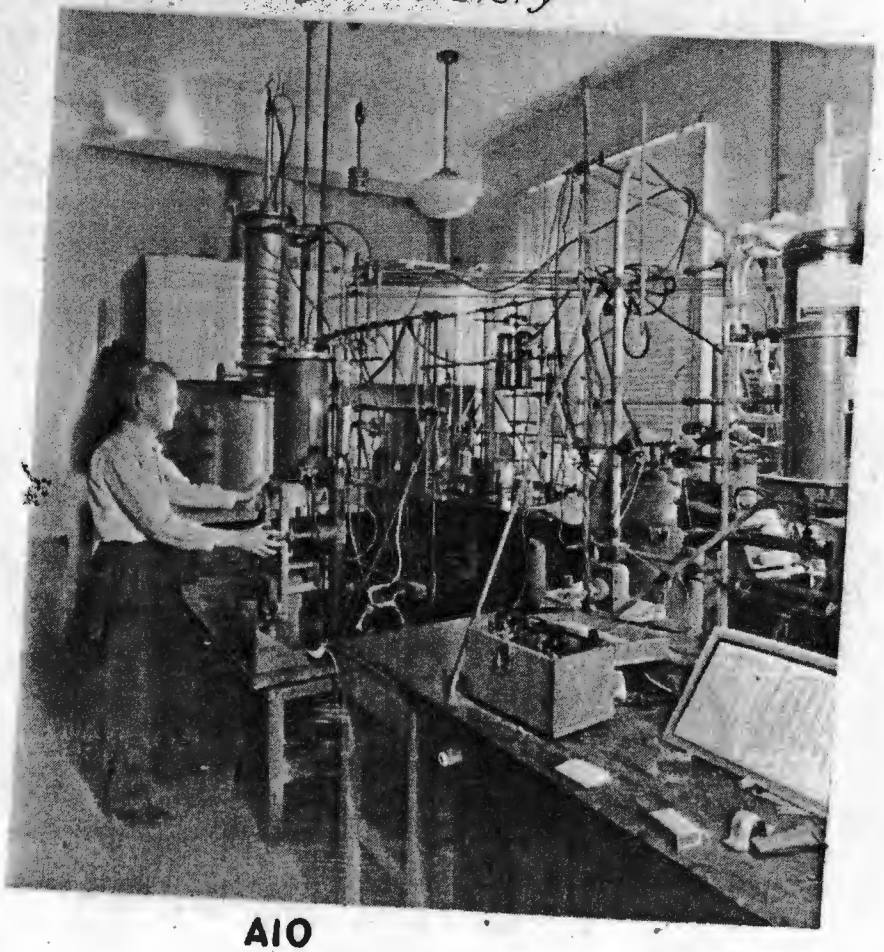

PLATE XVIII
Charge Vaporization Research

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MANHATTAN DISTRICT HISTORY

BOOK V - ELECTROMAGNETIC PROJECT

VOLUME 2 - RESEARCH

APPENDIX "B"

DIAGRAMS AND CHARTS

No.	Description
1	The Electromagnetic Method of Separating the Components of Uranium
2	The 1½-inch Area Map
3	Laboratory Personnel by Months (UCRL)
4	Tennessee Eastman Corporation Personnel at Berkeley Laboratory Personnel at Site X (Y-12)
5	Typical Organization Chart for University of California
6	Typical Organization Chart for the Research and Development Division of Tennessee Eastman Corporation
7	Typical Organization Chart for Brown University Research
8	Typical Organization Chart for Purdue University Research
9	Typical Organization Chart for Johns Hopkins University Research
10	Flow of Material in Electromagnetic Plant
11	Project 48 Expense (UCRL)
12	Chemical Research and Development Costs, Tennessee Eastman Corporation
13	Project Expense, Brown University
14	Project Expense, Purdue University
15	Project Expense, Johns Hopkins University

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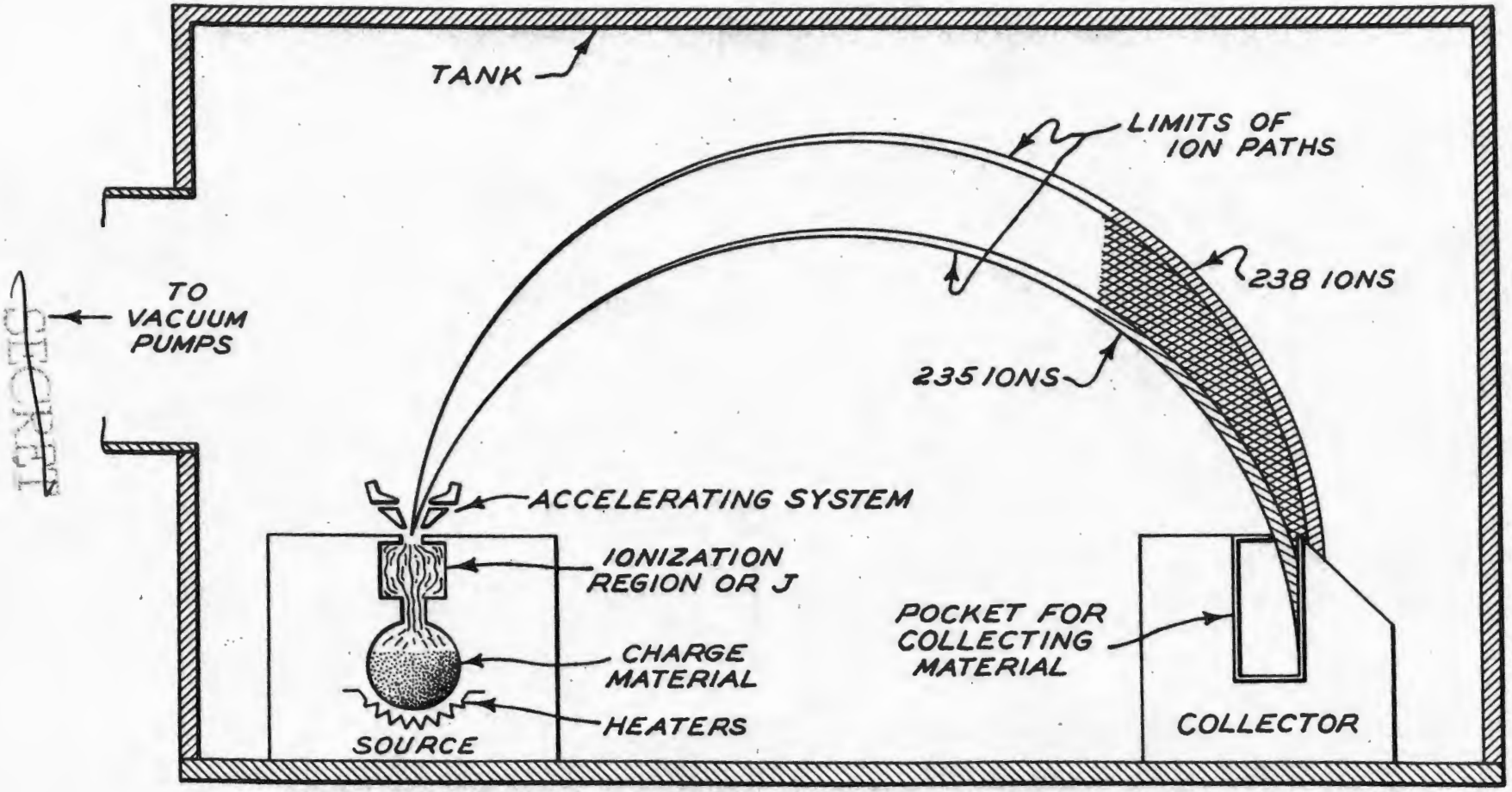
B1 The Electromagnetic Method of Separating the Components of Uranium

The isotopes start as a single solid beam from the source, but by the time they are midway between source and collector the two broad beams have already begun to diverge slightly. Cross-hatching is used in the diagram to represent the U-235 beam (hatch lines slope 30° down to right) and the U-238 beam (hatch lines slope 30° upward to right). The two beams intermingle in the double-hatched space. It may be noticed that, while the beams themselves are narrowed at the collector, the separation of the two beams is greatest at the collector. The narrowing of the beams is due to the focusing effect of the magnetic shims. The problem is to collect that free portion of the U-235 beam which does not coincide with the U-238 beam. The collector shown in the diagram will pick up a small portion of the U-238 beam.

THE E M METHOD OF SEPARATING THE COMPONENTS OF URANIUM



MAGNETIC FIELD PERPENDICULAR TO PLANE OF PAPER

FIG. 1

SECRET
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B1
FIG. 1

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[redacted] The 12½° Area Map.

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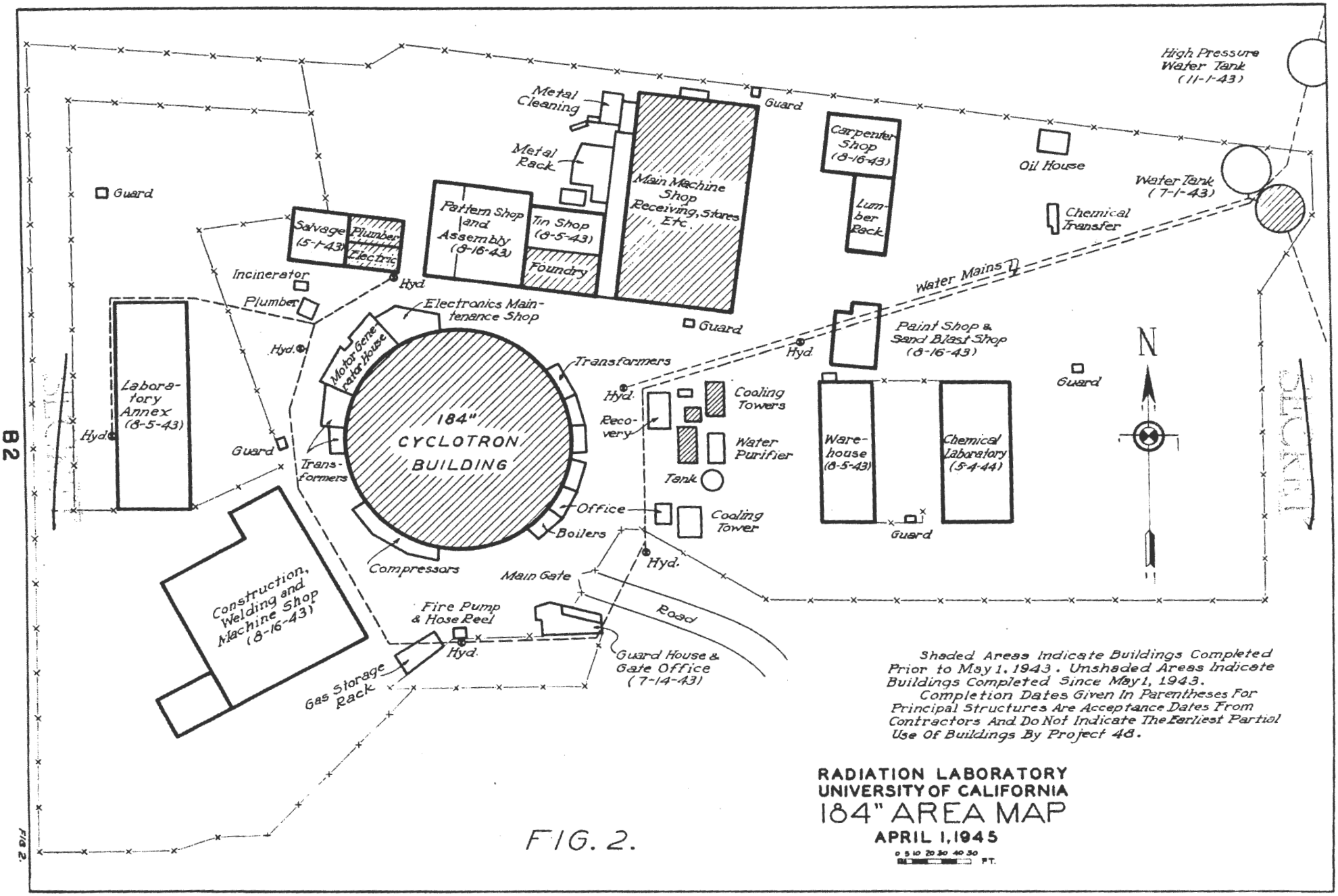

Shaded Areas Indicate Buildings Completed Prior to May 1, 1943. Unshaded Areas Indicate Buildings Completed Since May 1, 1943.
Completion Dates Given In Parentheses For Principal Structures Are Acceptance Dates From Contractors And Do Not Indicate The Earliest Partial Use Of Buildings By Project 48.

RADIATION LABORATORY
UNIVERSITY OF CALIFORNIA
184" AREA MAP
APRIL 1, 1945

0 5 10 20 30 40 50 FT.

FIG. 2.

B2

FIG 2.

~~SECRET~~

B3 Laboratory Personnel by Months (U.C.R.L.)

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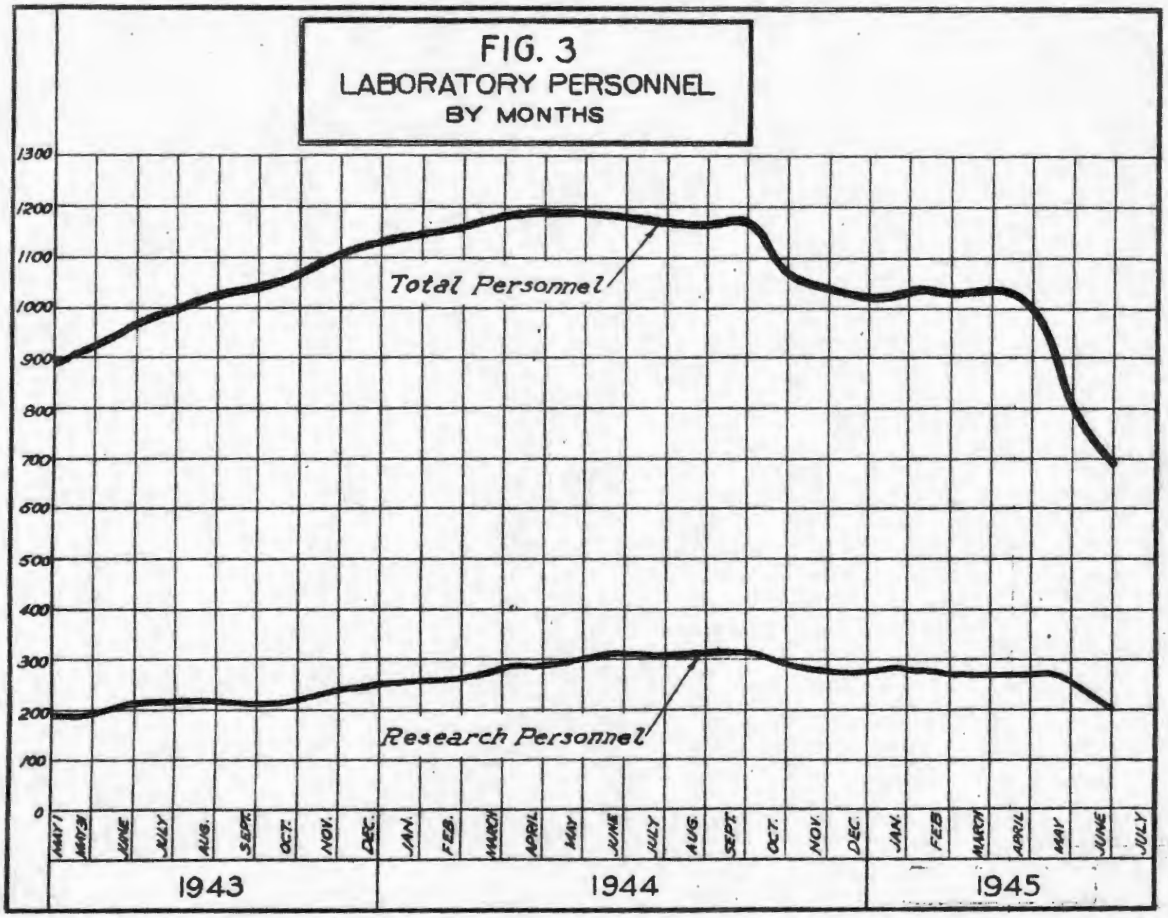

REPRESENTATIVE PERSONNEL DISTRIBUTION

	5-1-43	1-1-44	7-1-44	1-1-45	7-1-45
RESEARCH	192	243	303	269	203
ADMINISTRATIVE AND CLERICAL	142	142	193	172	137
PLANT OPERATION	194	245	218	167	100
SHOPS	141	223	238	240	120
PLANT MAINTENANCE	157	178	140	105	77
GUARDS AND OTHERS	65	93	85	66	52
TOTALS	891	1124	1177	1019	689

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b4 Tennessee Eastman Corporation Personnel at Berkeley.
Laboratory Personnel at Site X (Y-12).

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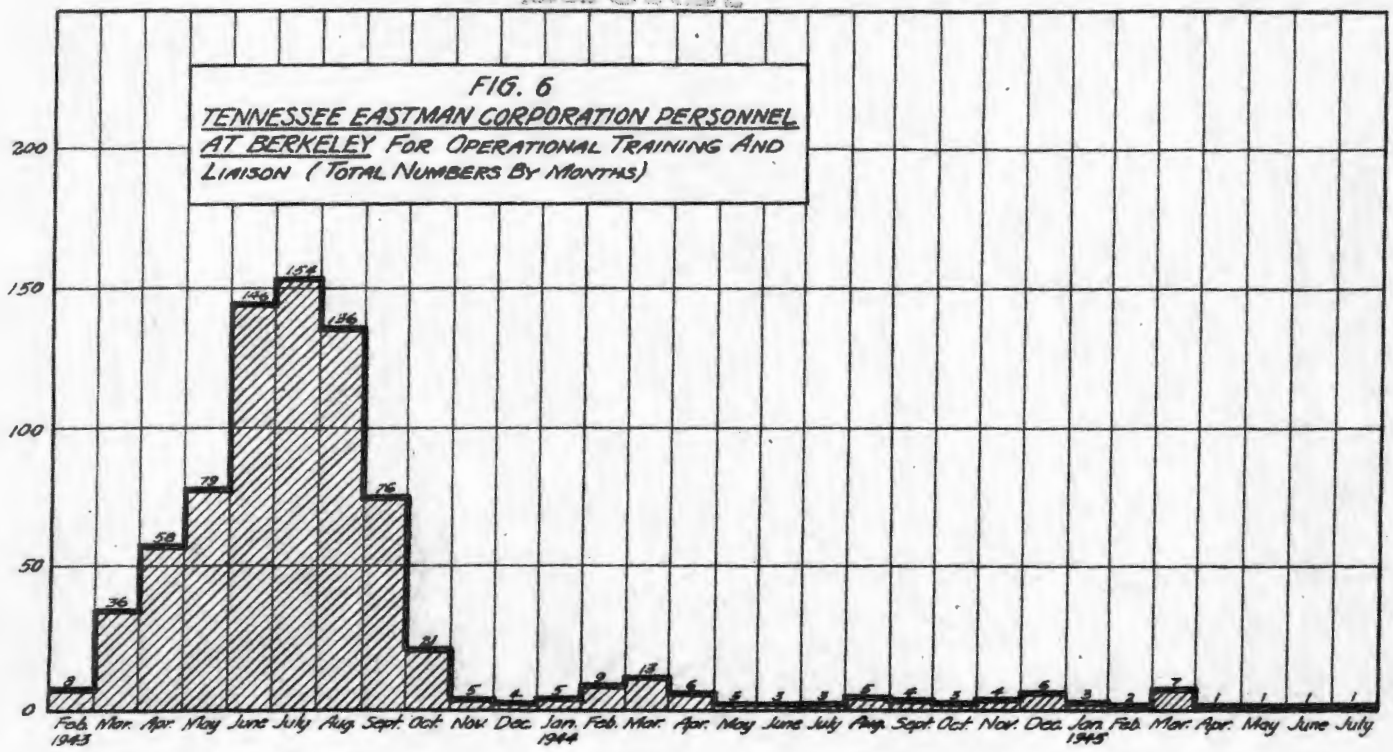
FIG. 6
TENNESSEE EASTMAN CORPORATION PERSONNEL AT BERKELEY FOR OPERATIONAL TRAINING AND LIAISON (TOTAL NUMBERS BY MONTHS)
200
150
100
50
0
9
36
58
79
146
154
136
76
21
5
4
5
9
13
6
3
3
3
6
6
5
4
6
3
3
7
1
1
1
1
Feb. 1943
Mar.
Apr.
May
June
July
Aug.
Sept.
Oct.
Nov.
Dec.
Jan. 1944
Feb.
Mar.
Apr.
May
June
July
Aug.
Sept.
Oct.
Nov.
Dec.
Jan. 1945
Feb.
Mar.
Apr.
May
June
July

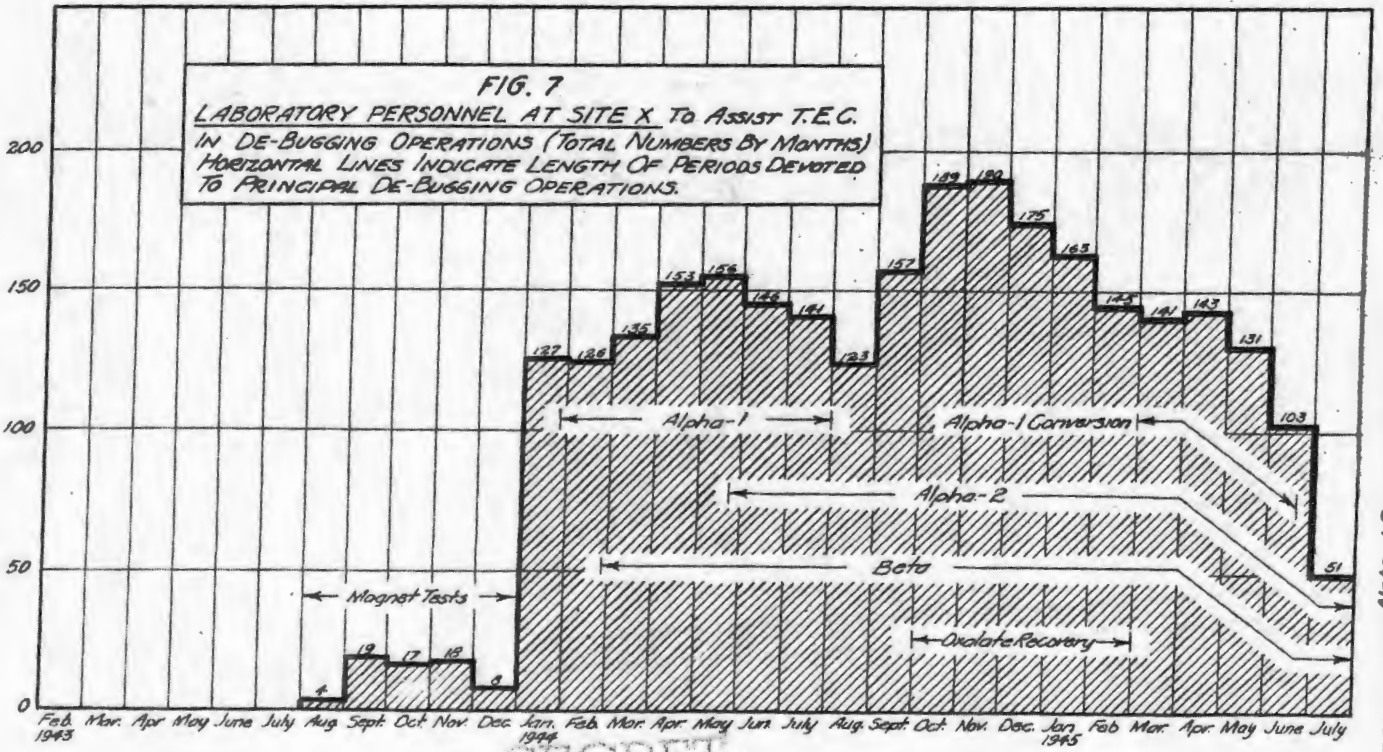
FIG. 7
LABORATORY PERSONNEL AT SITE X TO ASSIST T.E.C. IN DE-BUGGING OPERATIONS (TOTAL NUMBERS BY MONTHS) HORIZONTAL LINES INDICATE LENGTH OF PERIODS DEVOTED TO PRINCIPAL DE-BUGGING OPERATIONS.
200
150
100
50
0
4
19
17
18
9
127
126
135
153
156
144
141
123
157
189
189
175
163
145
141
143
131
103
51
Alpha-1
Alpha-1 Conversion
Alpha-2
Beta
Magnet Tests
Oxalate Recovery
Feb. 1943
Mar.
Apr.
May
June
July
Aug.
Sept.
Oct.
Nov.
Dec.
Jan. 1944
Feb.
Mar.
Apr.
May
Jun.
July
Aug.
Sept.
Oct.
Nov.
Dec.
Jan. 1945
Feb.
Mar.
Apr.
May
June
July

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B5 Typical Organization Chart for University of California.

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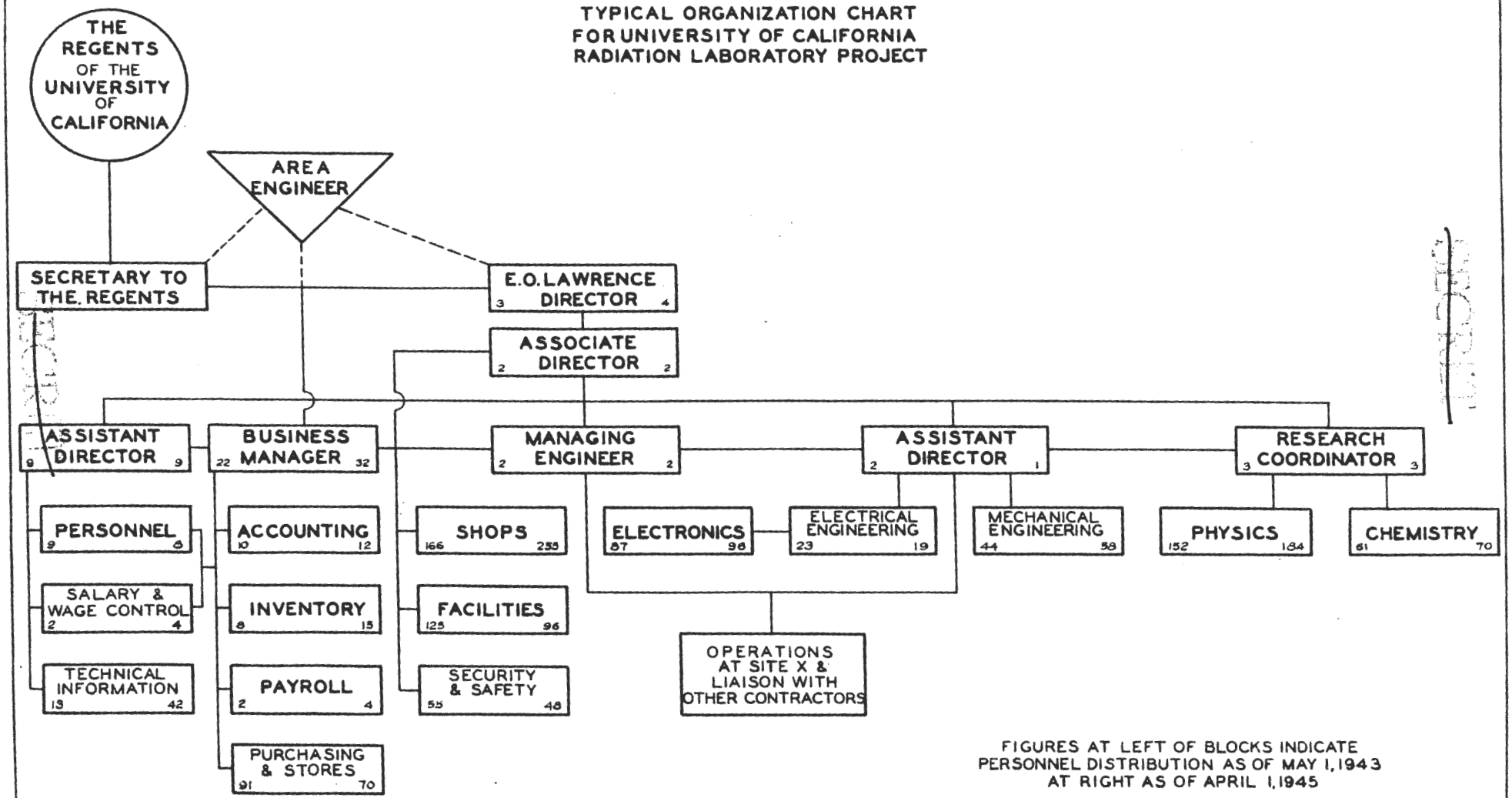
FIG. 5

TYPICAL ORGANIZATION CHART
FOR UNIVERSITY OF CALIFORNIA
RADIATION LABORATORY PROJECT

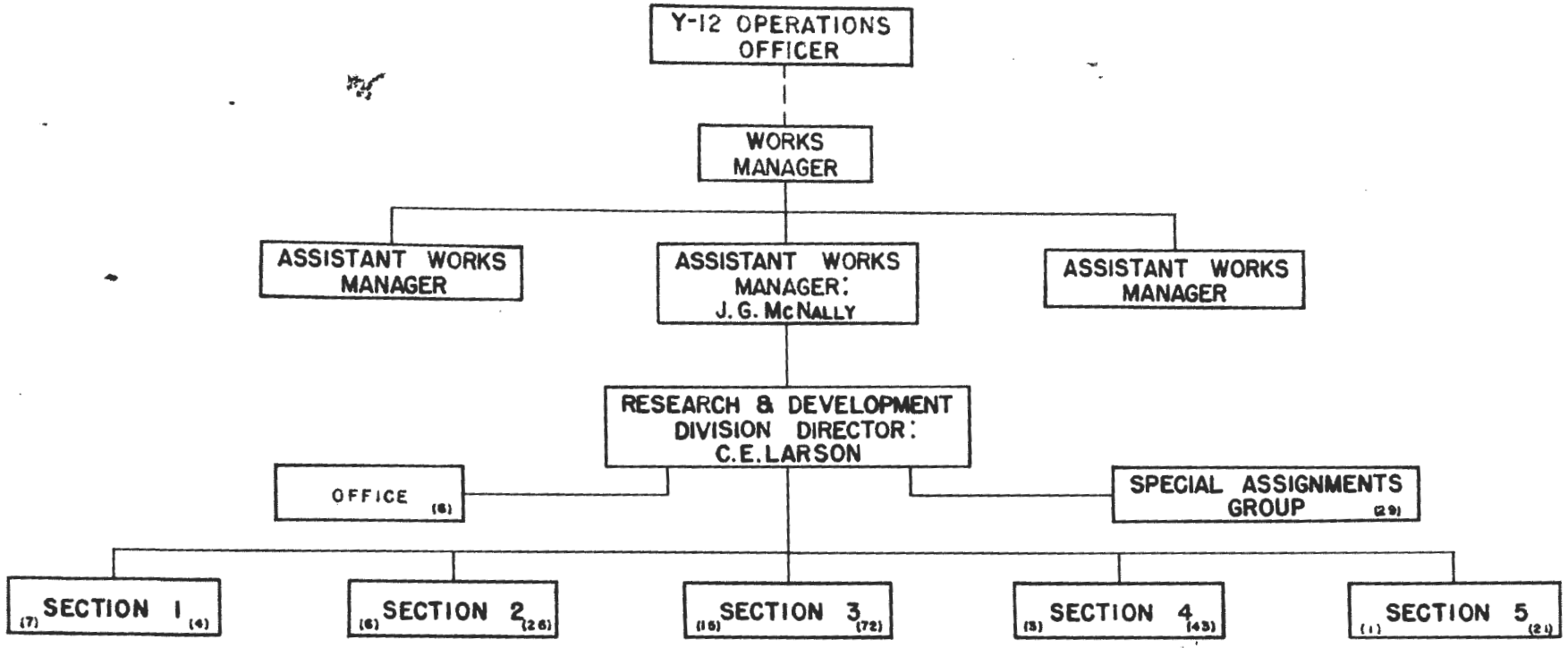


FIGURES AT LEFT OF BLOCKS INDICATE
PERSONNEL DISTRIBUTION AS OF MAY 1, 1943
AT RIGHT AS OF APRIL 1, 1945

FIG. 5

B6 Typical Organization Chart for the Research and Development Division of Tennessee Eastman Corporation.

TYPICAL ORGANIZATION CHART
FOR THE RESEARCH AND DEVELOPMENT
DIVISION OF TENNESSEE EASTMAN CORPORATION



FIGURES AT RIGHT OF BLOCKS
INDICATE NUMBER OF TECHNICALLY-
TRAINED PERSONNEL IN EACH SECTION.
FIGURES AT LEFT OF BLOCKS
INDICATE NON-TECHNICAL PERSONNEL.
ALL FIGURES ARE AS OF JUNE 30, 1945.

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57 Typical Organization Chart for Arms University Research.

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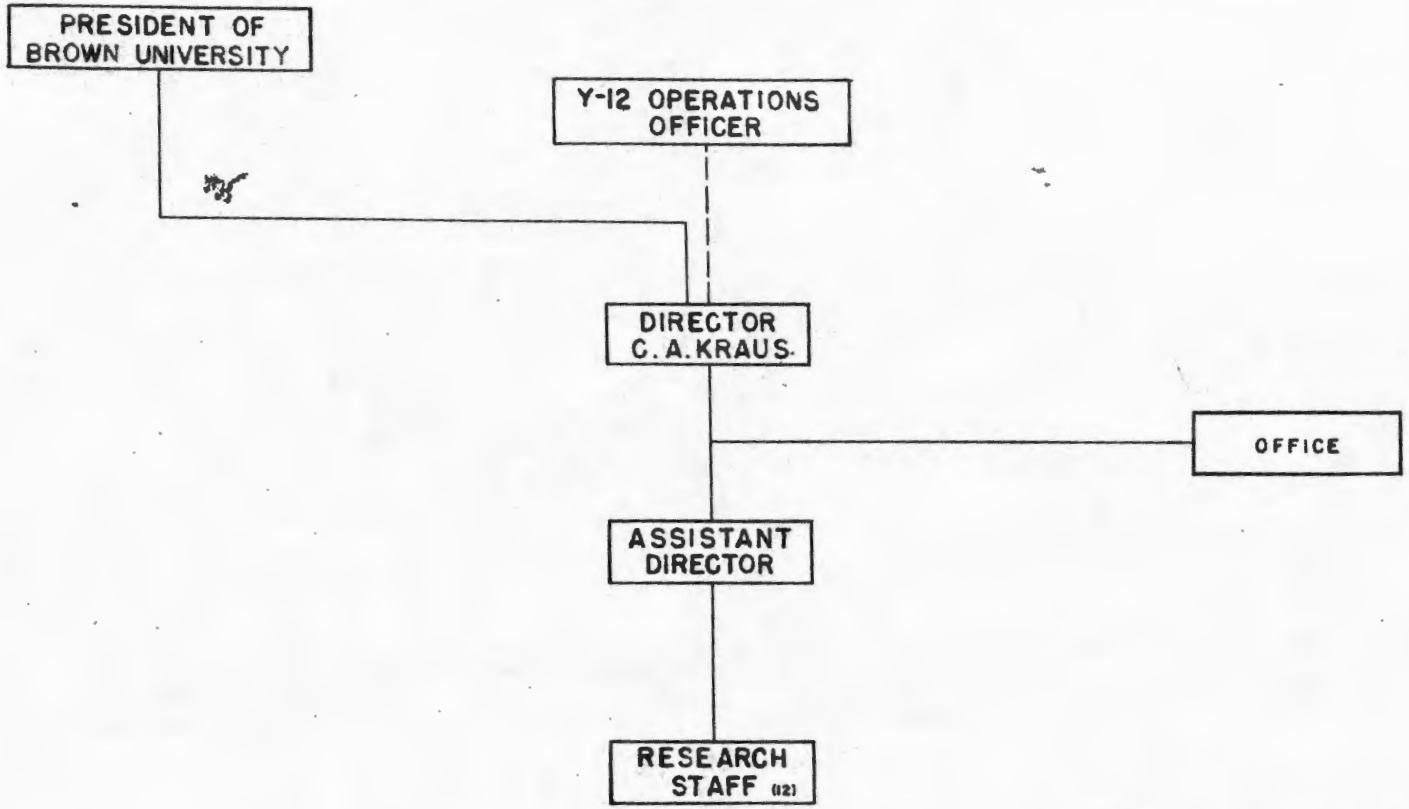
TYPICAL ORGANIZATION CHART FOR BROWN UNIVERSITY RESEARCH



FIGURES AT RIGHT OF BLOCKS INDICATE PERSONNEL DISTRIBUTION AS OF JUNE 30, 1945

~~SECRET~~

38 Typical Organization Chart for Purdue University Research.

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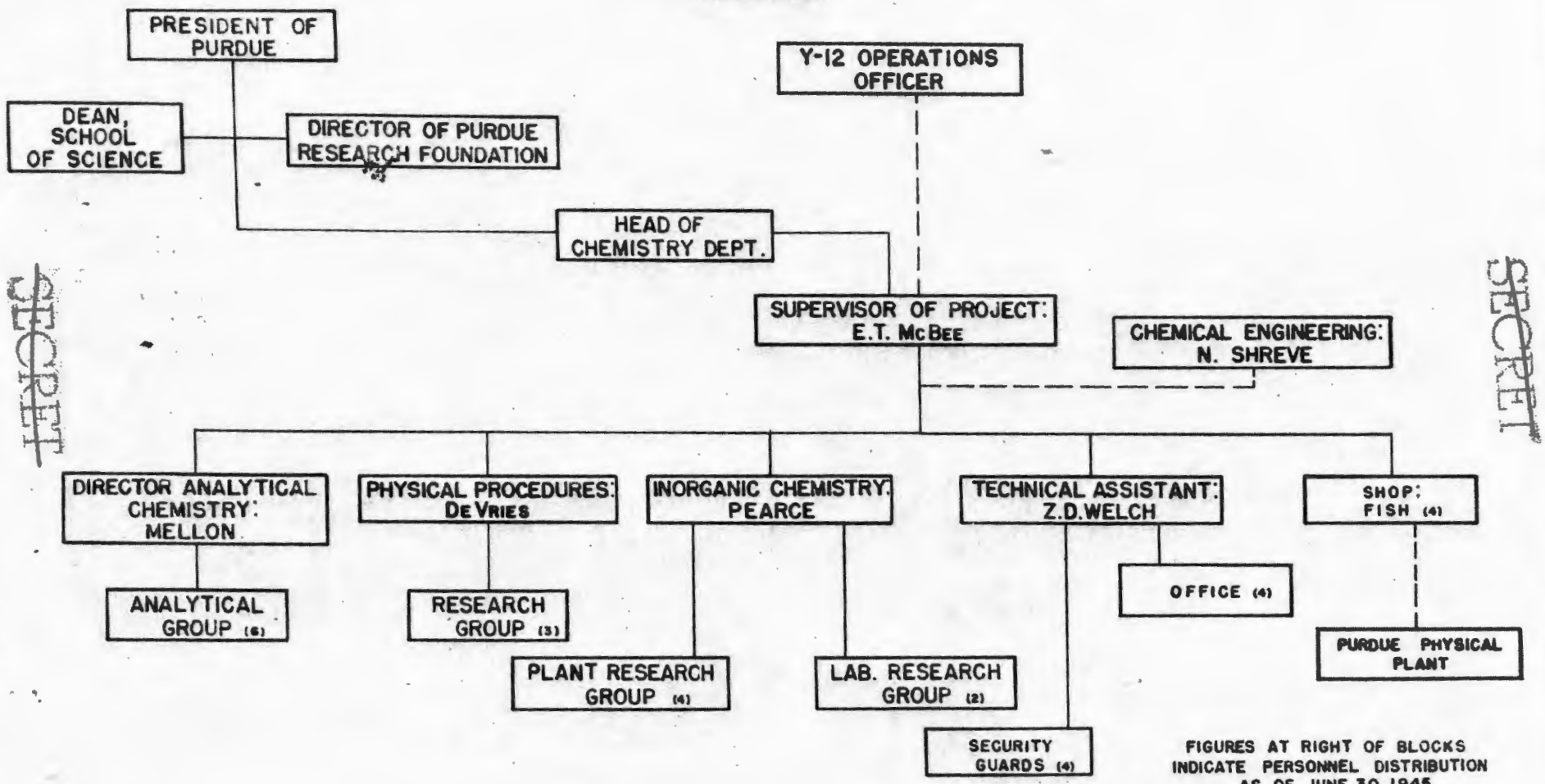
TYPICAL ORGANIZATION CHART FOR PURDUE UNIVERSITY RESEARCH



FIGURES AT RIGHT OF BLOCKS INDICATE PERSONNEL DISTRIBUTION AS OF JUNE 30, 1945.

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59 Original Organization Chart for Johns Hopkins University Research.

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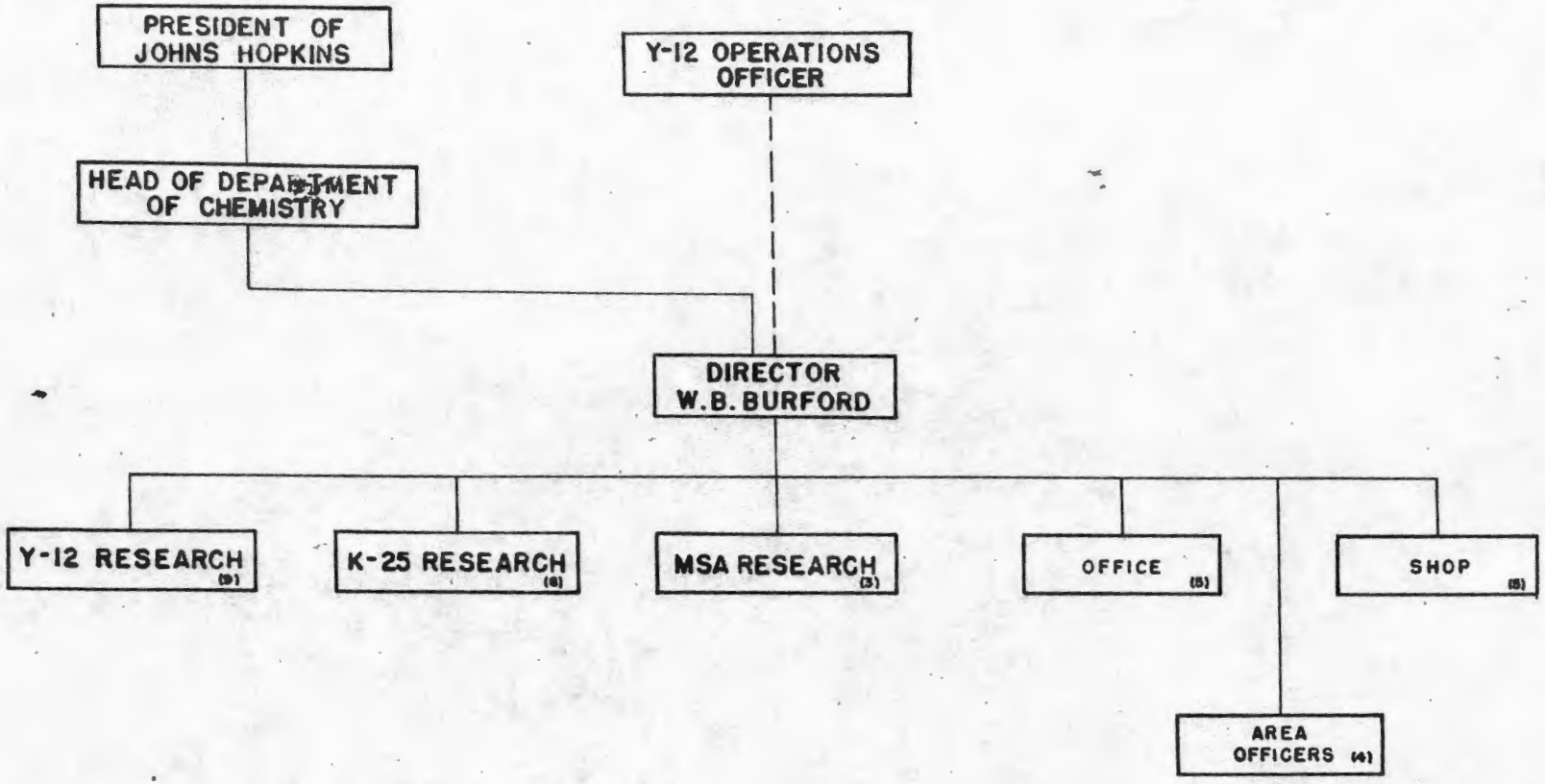
TYPICAL ORGANIZATION CHART FOR JOHNS HOPKINS UNIVERSITY RESEARCH



FIGURES AT RIGHT OF BLOCKS INDICATE PERSONNEL DISTRIBUTION AS OF JUNE 30, 1945.

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B10 Flow of Material in Electromagnetic Plant.

Raw feed materials, uranium trioxide (UO_3) and uranium hexafluoride (UF_6) are converted to charge material, uranium tetrachloride (UCl_4), in Alpha Chemistry. This UCl_4 is separated in the Alpha mass spectrographs to give a metallic product enriched to approximately 10% U-235. The unseparated UCl_4 in the mass spectrograph, such as material splattered on the sides of the machine, is cleaned from the machine, and recycled through Alpha Chemistry.

The Alpha product (contaminated uranium) is purified and converted to UCl_4 charge material by Beta Chemistry. At this point enriched UF_6 from K-25 is also converted to UCl_4 charge material. The Beta mass spectrographs separate the final enriched material and the unseparated uranium tetrachloride is recycled through Beta Chemistry.

Final Chemistry receives the Beta Product (contaminated uranium metal) from the mass spectrographs and converts this material to product ready for shipment in the form of uranium tetrachloride.

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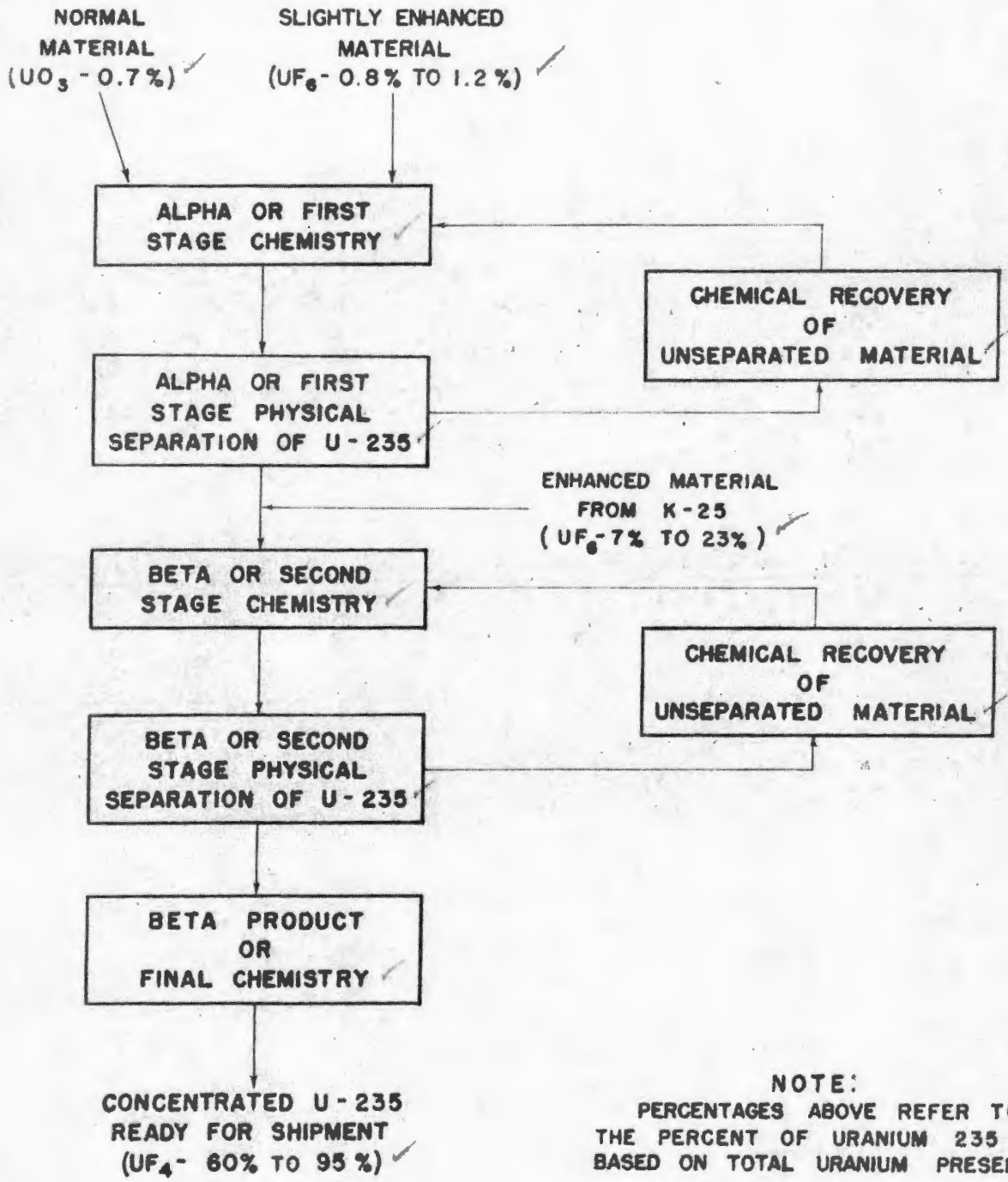
FLOW OF MATERIAL IN ELECTROMAGNETIC PLANT



NOTE:
PERCENTAGES ABOVE REFER TO THE PERCENT OF URANIUM 235 BASED ON TOTAL URANIUM PRESENT.

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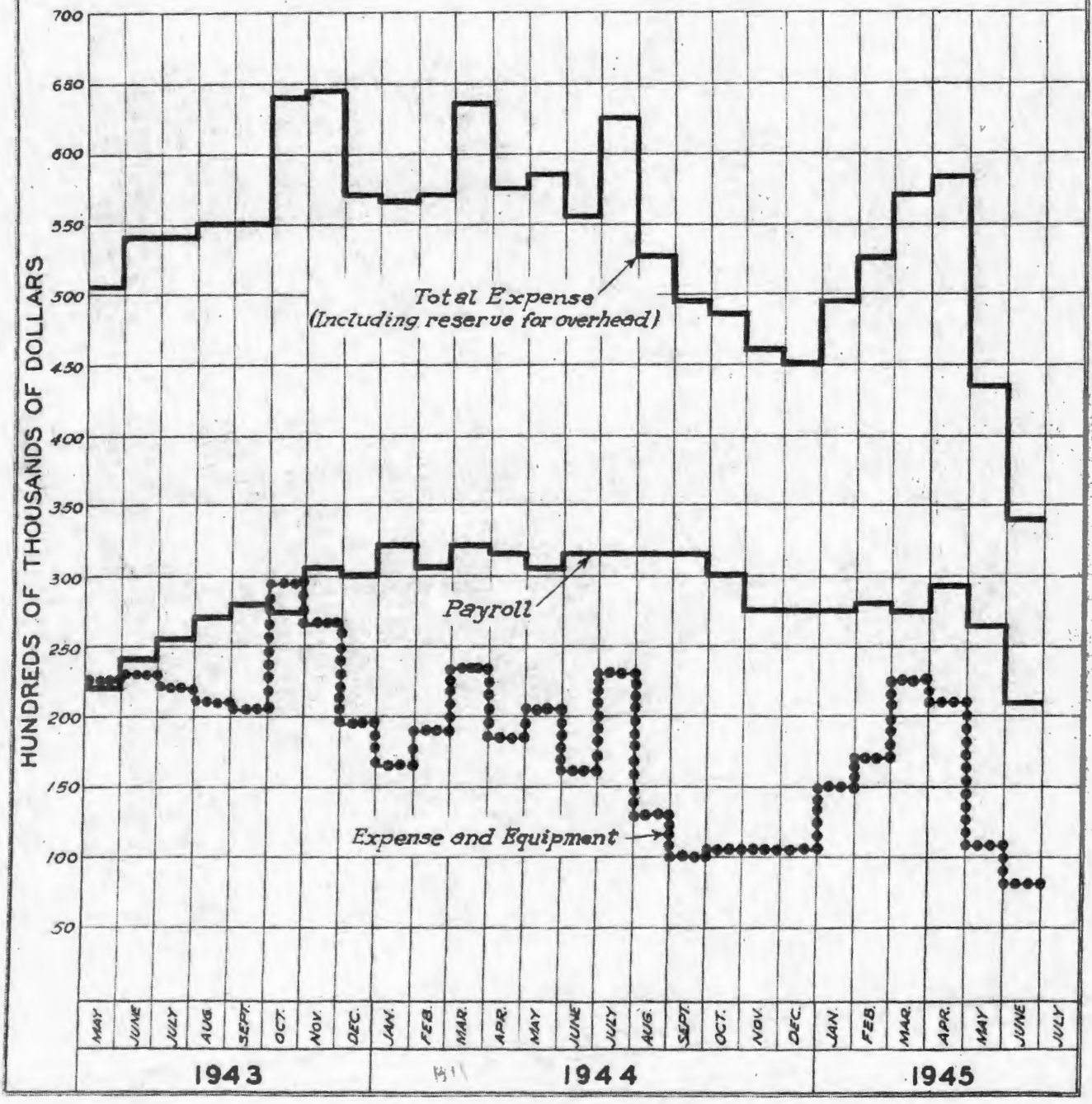
B11 Project 4B Expenses (UCRL).

~~SECRET~~

FIG. 4
PROJECT 48 EXPENSE
ESTIMATED BY MONTHS TO
THE NEAREST $ 5,000



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M2 Chemical Research and Development Costs, Tennessee Eastman Corporation.

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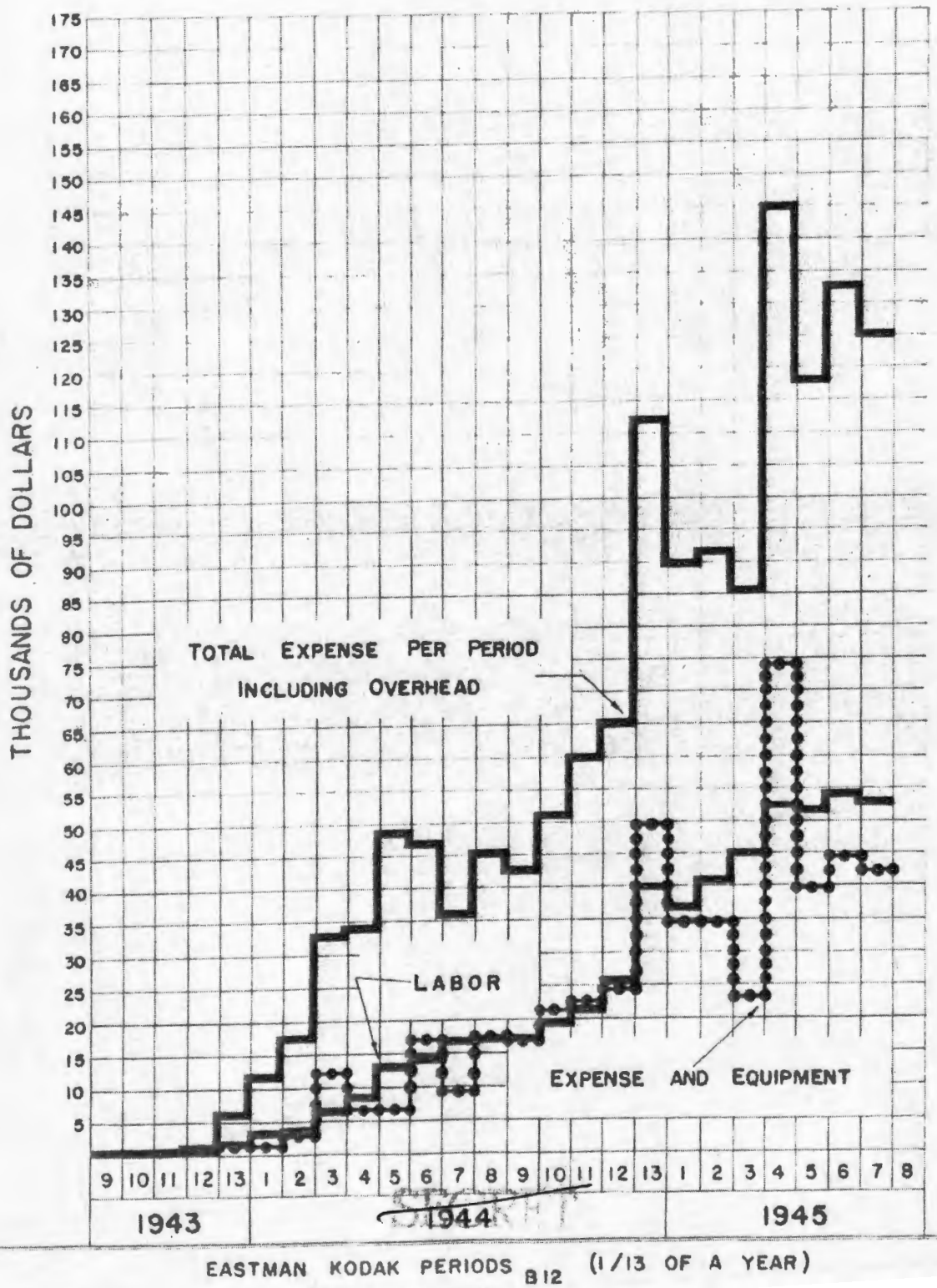
TENNESSEE EASTMAN CORPORATION

CHEMICAL RESEARCH & DEVELOPMENT COSTS

CONTRACT W-7401 ENG-23



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B13 **Project Expense, Brown University.**

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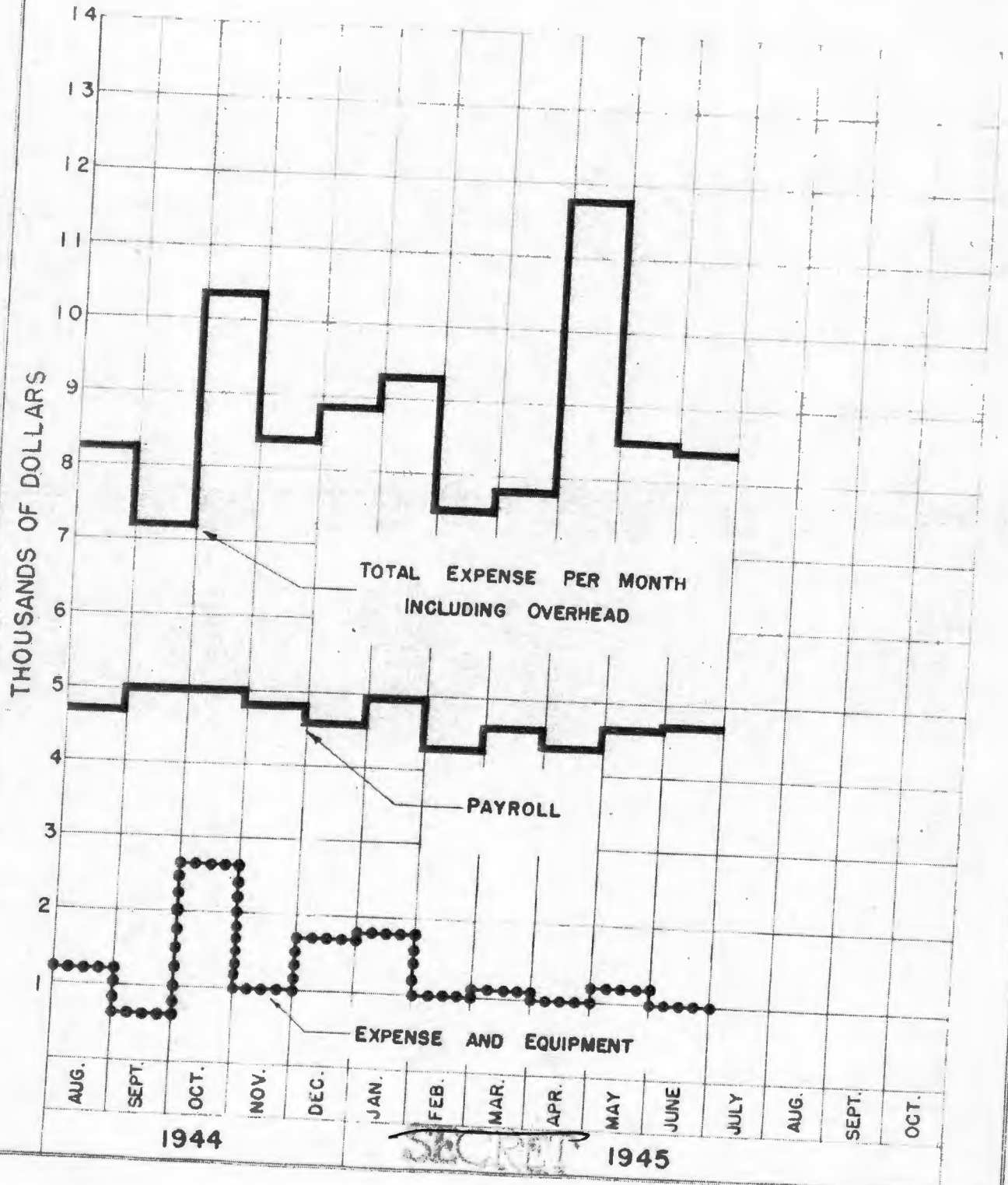
SEC

BROWN UNIVERSITY

PROJECT EXPENSE

CONTRACT W-7405 ENG-73



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B14 Project Hermes, Purdue University.

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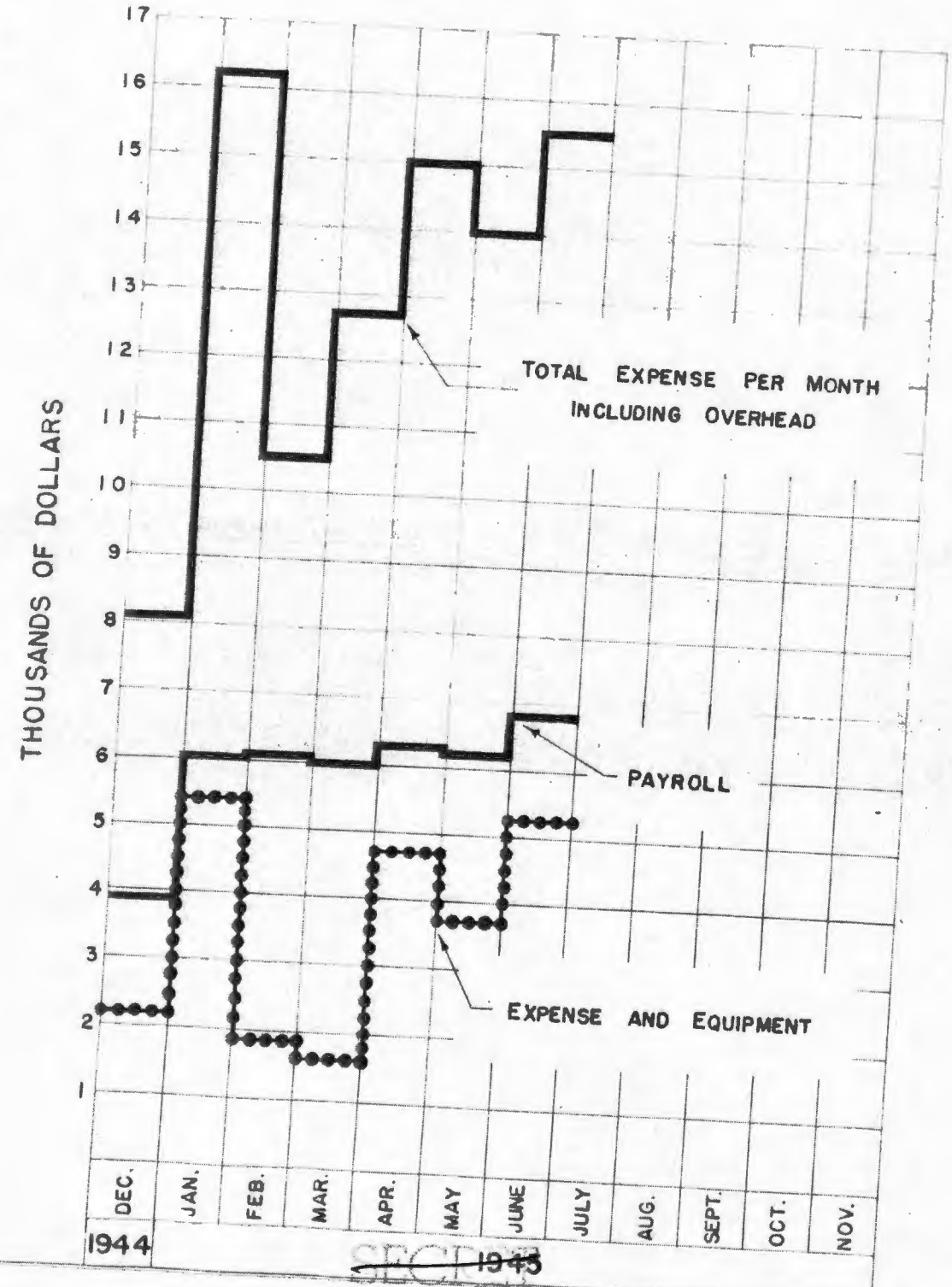
PURDUE UNIVERSITY

PROJECT EXPENSE

CONTRACT W-7405 ENG - 74



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B15 Project Express, Johns Hopkins University.

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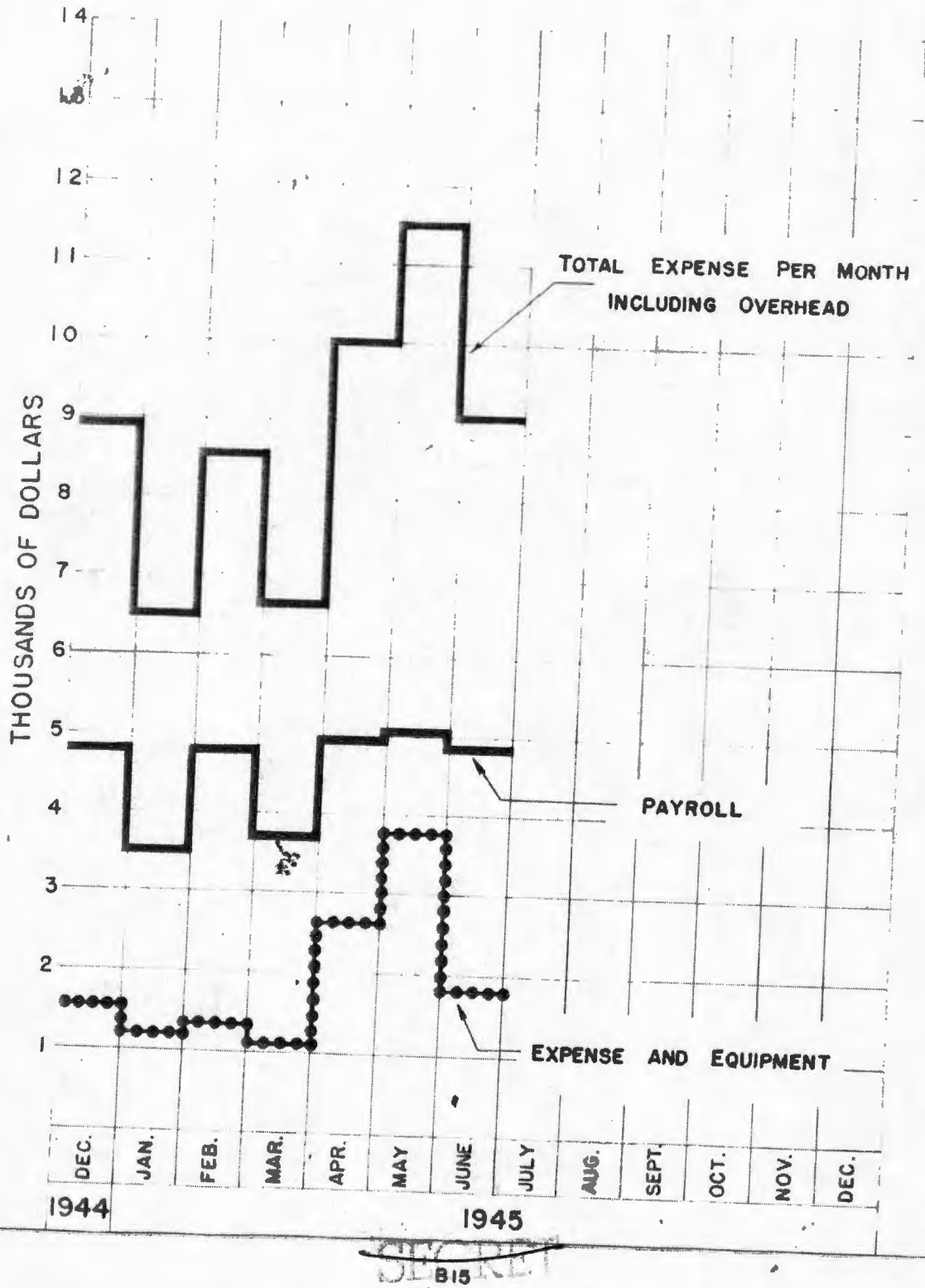
JOHNS HOPKINS UNIVERSITY

PROJECT EXPENSE

CONTRACT W-7401 ENG-43



B15

This Document contains ____ Pages.
This is copy 3 of 6 Series A

5404

JAN 4 1946

MONTHLY REPORT - CHEMICAL GROUP

September-1942

I. Preparation of Tuballoy Halides.

Work early this month centered principally on the tetrabromide. Recently, however, it appears that the tetrabromide is not as suitable as the tetrachloride. Further work on the bromide is therefore dependent on additional tests on the hill.

Tuballoy tetrabromide is much more difficult to prepare than the tetrachloride, as shown by our experience, and as reported by the Bureau of Standards.

The only method found satisfactory is according to the reaction:

T_3O_8 (or T^0_2) + C + Br_2 $\longrightarrow$ TBr_4 + (CO + CO_2 + $COBr_2$)

Our experiments (see Fig. 1) extended only to about 800°C. The experience of the Bureau of Standards is that a satisfactory rate is attained only at 1000°C. At 800°C, we made only 50-75 gm in 3 hours. The Bureau at 1000° was preparing a kilogram in 8 hours. It was found essential, in obtaining a reactive carbon-tuballoy oxide mixture, to proceed by charring a sugar-oxide mixture. Merely mixing carbon, even lamp-black, with the oxide, gave a charge reacting very slowly with bromine.

A number of improvements in the preparation of the bromide have been planned, including the possibility of conversion of the chloride to bromide. If further work on the hill indicates the bromide is desirable, these will be continued.

On the preparation of tuballoy tetra-chloride, much greater progress has been made. We now have several methods which can turn out a kilogram or

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more per run. The principal problems in this line at present are: to determine the most satisfactory materials for constructing equipment, and to work out practical details for regular production. Short summaries of the various methods are given below.

Production from carbon-tubalioy oxide mixture and chlorine. (See Fig. 2)

This also is essentially a high temperature reaction, requiring temperatures above 800°C. It also requires preparation of the charge by charring a sugar-oxide mixture. The product, however, is very pure (99 + %) and separated from the charge. One short run using small amounts gave 150 g of product.

Production of tubalioy tetrachloride from tubalioy oxide and sulphur mono chloride.

This method was used by the Bureau of Standards and by the commercial supplier. It proceeds according to the reaction:

$$T_3O_8 \text{ (or } TO_2\text{)} + S_2Cl_2 \longrightarrow TCl_4 + (SO_2 + S + \ldots)$$

(See Fig. 3)

As run by the Bureau of Standards, this was carried out at 1000°C. Our experiment was also done at this temperature, but it is probable it will go at considerably lower temperatures. One six-hour run by this method gave us 1675 grams of product, (1400 grams 96% plus) from a 2000 gram charge of T_3O_8 in 5 hours. A principal difficulty is the removal of sulphur (or sulphur compounds) from the product.

Production of tubalioy tetrachloride from tubalioy oxide and carbon tetrachloride.

The upper temperature limit on this reaction is fixed by thermal decomposition of the carbon tetrachloride to carbon and chlorine at about 750°C. The reaction apparently proceeds in two or more ways, of which the first occurs at lower temperatures than the rest:

$$TO_2 + 2CCl_4 \longrightarrow TCl_4 + 2COCl_2$$

$$TO_2 + 2COCl_2 \longrightarrow TCl_4 + 2CO_2$$

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Our first runs by this method were made at 650-700°C., after short period above 750° had shown the production of considerable carbon. They gave more than a kilogram each of product analyzing 99.5% TCl_4. The product was the best looking of any yet made, and was chiefly in the form of long black lustrous crystals. A small amount (same purity) was in the form of a dense very dark green powder.

In the two runs above, there was some difficulty in separating the product from the charge, because of the slow rate of distillation at 650°C. The attempt was therefore made to convert the oxide to the chloride in place, with distillation at all, at a somewhat lower temperature, and to subsequently distill the product from the unreacted charge at a much higher temperature, in the absence of any carbon tetrachloride. A run was then made at 450°C, using 500 g of tuballoy oxide. After three hours, the run was stopped. The material in the tube appeared entirely converted to chloride, consequently, instead of distilling it off, it was removed for analysis. It should be mentioned that one of the principal obstacles to the preparation of a pure product is the difficulty of removing it from any surface on which it has condensed, or on which it has been melted. The adhesion is so great that the container is apt to be broken, and in any event the time consumed gives plenty of opportunity for hydrolysis by the moisture unavoidably present.

In the low-temperature run just described, however, the product was in the form of fine, separate crystals, with a small amount of unreacted oxide (in lumps) which could be completely removed from the tube by pouring it out. A material balance of the charge and product gave a value of 90% conversion to the chloride. The crystalline material analyzed 93% TCl_4; the small amount of lumps were 77% TCl_4.

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Preparation of tuballoy tetrachloride from tuballoy carbide and chlorine.

Some small-scale experiments on this preparation have been made. It appears that the carbide is completely converted to the chloride at low temperatures, (about 350°), leaving only a small residue of light carbon particles. Future work on this problem is planned as soon as more tuballoy carbide is available.

Summary of work on preparation of tuballoy chloride.

A number of methods of preparation have been developed, all of which proceed at a satisfactory rate and nearly to completion. Regular production of a satisfactory product depends on a number of factors, now being studied:

1. Materials to withstand the temperatures and corrosive conditions encountered. Some that are under investigation are: quartz, silicon carbide, porcelain, graphite, and pyrex.

2. Condenser materials not wet by TCl_4. Graphite is being investigated.

3. Satisfactory separation of charge and product. This can be done by distillation at a high temperature, or by a substantially complete conversion without distillation.

4. Choice of a one or two-step process. It is possible that satisfactory yields of 99 + % material can be produced in one step. However, it may be simpler and faster to make 90% material and subsequently to purify it by distillation in vacuum.

Purification of tuballoy halides:

Work on this problem has been almost entirely confined to distillation methods. The vacuum still, which has been in use, has been redesigned to prevent any mixing of the crud and the distillate, and to provide a more satisfactory method of heating (see Fig. 4). Recent tests on the new still show it to be quite satisfactory. The new still will take a kilogram of charge,

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and complete the distillation in less than 3 hours.

Another method of distillation is in a stream of nitrogen (see Fig. 5). This has the advantage of not requiring a vacuum vessel good at high temperatures, and of permitting condensation at higher temperatures to give larger crystals. Several runs made with small amounts have shown that this is a suitable method.

II Recovery of Unused Charge

Organization and Purposes

At the start of last month, it became evident that adequate procedures for efficient recovery of the source material would be required to make the general process feasible on a large scale. It was felt on grounds of economy that recovery of material both on the first (crude) run as well as the second (enrichment) run might also be desirable. The writer, at the request of Prof. Jenkins, then organized a crew including Mr. Schmidt, Mr. Kilner and himself for the purpose of investigating all types of recovery procedures which might be contemplated. This report chronicles the experiments performed and conclusions drawn to date.

Methods of Recovery

The procedures to be devised depend on the nature of the tubalioy deposit, the portion of the vacuum chamber from which the deposit is obtained, and the major contaminants introduced by the metals used in the construction of the apparatus. One may classify recovery procedures as follows:

1. Dry methods.
2. Wet methods.
3. Electrolytic methods.

Each of these has its special advantages and it has been the task of the Recovery Crew to determine the exact conditions under which each is feasible.

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Experiments on Recovery from Copper and Iron using Wet Methods

The first experiment was performed with the 39" can. The purposes here were to ascertain how the tuballoy distributed itself under conditions of a cold arc source and how quantitatively the source material could be recovered using water and mechanical action (i.e. scrubbing). The geometry was as indicated diagrammatically below (Fig. 6). A "C" was constructed on a radius (inside) of 1' and with an aperture of 6" x 6". The inside of the "C" was lined with 1 mil Cu foil. Foils were placed immediately above and below the source shield to collect all material falling in the space between the shield and the "C". To ascertain how much tuballoy got past the "C" and distrubuted itself around the vacuum can, the liquid air trap was covered with foil also. No foils were used on the collector. A charge of 88.6 grams was placed in the reservoir. When the run was finished, the can was opened and the distribution first ascertained visually. It could be seen that practically all the material (approx. 99%) was collectible form the ion source and shield and the region between the shield and the "C". No more than approx. 0.2 grams of tuballoy was in the "C". The inside of the "C" showed no signs of heavy ion bombardment, a smooth, black coating of the tuballoy metal being found spread evenly throughout the whole inside of the "C". Less than 1/10 milligram of tuballoy deposited on the liquid air traps showing that the "C" collected all the tuballoy not remaining in the source region. The collector showed some pitting and a small deposit of tuballoy about equivalent to what was in the "C". Since all the material seemed to remain in the ion source region, the source, shield and slits were carefully cleaned with water and a brush. The recovery data is listed below:

Source and Shield -	63 grams
Slits	21 grams
Reservoir	6 grams
Total	90 (± 5) grams

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It is seen that by this method, all the tuballoy regardless of its state of combination can be recovered from copper or brass by vigorous mechanical action and washing. It was noted that while little effort was required to remove the deposit from a graphite surface (slits), the coating on copper proved very tenacious. In a separate experiment pure tuballoy chloride was rubbed into copper and allowed to stand in air. Hydrolysis and reaction on the copper by the acid formed resulted in a closely adhering, chemically combined coating of the tuballoy chloride (probably as tuballyl chloride or oxide) with the copper. Only acid (approx. 6 N HCl) sufficed to remove this coat completely.

Since it appeared that copper might be the major contaminant and complication in the recovery of the tuballoy, methods for the separation of copper and tuballoy were investigated, keeping in mind that both large and small scale procedures might be needed.

The first type of procedure tried both for ascertaining possibilities for recovery as well as separation of tuballoy from copper was electrolysis. A preliminary experiment showed that to get high current efficiencies and smooth deposition of copper, a rotating electrode was required. The apparatus was built incorporating a large rotating copper cathode with variable speed of rotation and a sheet platinum anode. The protocol of a typical electrolysis run is given herewith:

Equipment: 1 Platinum Electrode (2" x 3")
1 Rotating Copper Electrode
(3.104 cm diameter, 6.3 cm long,
area = 0.6140 dm^2)

Reagent: Solution containing 1.13 mg tuballoy / cc and 40 mg copper / cc as chloride.

Electrolysis: 100 cc of solution adjusted to pH = 1 used.
Cathode rotated at 165 r.p.m.; current = 0.5 amperes
Voltage held below 2 volts.

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The pH was measured with a glass electrode as the electrolysis proceeded and was used to determine how quickly the copper was being removed. The pH dropped from 1 to 0.15 in 2 hours and in 3 hours, practically all of the copper color in solution was discharged. No tuballoy plated out. Current-voltage curves for pure copper solutions when compared with solutions containing both tuballoy and copper in the proportion indicated above showed that practically none of the current was carried by the tuballoy. Only by running the voltage up above 4 volts could one obtain some tuballoy oxide deposit on the cathode and this went back into solution when the current was turned off. Analysis of the completed run above showed 97% recovery of the tuballoy in solution whereas all of the copper was plated out. Much data on the voltage-current relations for various Cu-T solutions were obtained.

It can be concluded that one may remove tuballoy incorporated with copper into solution by electrolysis and afterward plate out the copper without plating out the tuballoy (because of the high over-voltage required to bring down tuballoy as the oxide at the cathode.)

The electrolytic setup was also found useful by the analysts for determination of the 4-valent tuballoy in the presence of the 6-valent form, as well as for a variety of other analyses and was turned over to them at the conclusion of these experiments.

Another method which suggested itself for use in separating tuballoy from copper was based on the selective absorption exhibited by activated alumina for metal ions. Chromatographic columns (i.e. long cylindrical tubes packed with alumina) were set up and the absorption characteristics of some laboratory alumina investigated. In the first experiment, alumina powder was slowly dropped through a long thin column of solution containing Cu and T thus

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reversing the usual chromatographic procedure in which the solution is percolated through the alumina. (This procedure was suggested by an article in Science, Vol. 96, 212.) Due to the heterogeneity of the alumina particles, variable speeds of dropping were encountered and the method proved impractical. Hence, recourse was had to the usual procedure of packed columns. In another experiment, 5 cc of a solution containing 9.70 mg T/cc and 10.0 mg Cu/cc (pH-2.95) were run slowly through a column of alumina packed into 3 mm glass tubing, using the vacuum of the water aspirator to pull the solution through the column. The tuballoy was completely absorbed as a green band on the alumina, the copper appearing below the tuballoy as a blue coloration. It proved possible by washing the alumina column to completely wash out the copper band while leaving the tuballoy still absorbed on the alumina.

The experiment was repeated using larger tubes and with the object of obtaining quantitiative data. It was found that one could effect 100% T recovery on the column while practically all the copper washed through. After the copper had been removed, the alumina was expelled from the tube and washed with 1 N HCl, whereupon all the T was dissolved off the alumina so that a complete separation of the T from the Cu was effected. The experiment was extended to iron and similar results obtained although separations were not as sharp as for the Cu-T couple. The iron is absorbed by the alumina first and then the tuballoy, so that by cutting the column into an iron and a tuballoy portion and eluting with acid, a separation is made. The effect of pH on these separations is being determined. In addition other absorbents, such as 8 - hydroxy quinoline, are being tried.

Attempts at separation by partition in organic solvents have yielded no simple process.

The chromatographic experiments have shown that additions of activated alumina to a solution containing Cu and T results in the quantitative removal

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of the tuballoy by absorption. This is the basis for a simple recovery process for tuballoy in the presence of copper.

Further experimentation on separation of tuballoy from various metal ions have been postponed until a decision is reached on the nature of the metallic coating to be used in the vacuum chamber.

Experiments with Stainless Steel

The tenacity of the tuballoy deposit on copper requiring vigorous rubbing to remove the last traces of tuballoy from a copper surface together with the complications introduced by the large amount of copper ion mixed with the tuballoy have dictated the use of a more resistant and less reactive metal surface for the vacuum chamber and parts exposed to ion deposition. Hence, foils of stainless steel have been exposed to tuballoy ion currents in both the 37" outfit and Tank "1 on the hill. It is found, as expected, that little or no bonding occurs due to chemical reaction. Washing with hot water suffices to remove all but a negligible portion of the tuballoy from the stainless steel surface. What is left after washing with water is found to be mainly sputtered copper and this can be removed by dipping in 0.1 N HNO_3 which does not attack the stainless steel. Similar experiments are contemplated with monel metal. Much data on the composition and corrosiveness of stainless steels of various grades have been compiled and all the indications are it will be desirable to use stainless steel for all surfaces exposed to tuballoy or tuballoy chloride deposition.

Conclusions

Since the plan at present is to heat the entire ion source region so that practically all the tuballoy deposits in the "C" or vacuum chamber, it appears

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that the recovery can be made from stainless steel surfaces almost entirely. If care is taken to keep the deposit dry when opening the vacuum chamber to air, the tubalioy can for the most part be scraped out as the dry salt and perhaps recovered by a simple distillation. The ion source containing little of the tubalioy can be cleaned with acid or water and the tubalioy recovered, if desired, by the methods outlined in the preceding pages. The tubalioy deposit not removed by scraping of the stainless steel can be cleaned off as often as necessary by a wet method, (either washing in water or dilute acid dip). A large air drying unit is now being obtained for further experiments with dry air in the 37" cutrit have shown the desirability of such a unit.

It is considered that recoveries very close to 100% of the sputtered tubalioy can be made with the methods now at our deposal. This will be tested as soon as a model of the setup on the hill (H1) can be constructed for use in the 37" magnet.

Submitted by J. M. Carter (Part I)
M. D. Zumwalt (Part II)
[illegible] (Part III)

III Analytical Methods

Organization activities of the Department consisted of assembling equipment and reagents in such form as to facilitate making of rapid accurate analyses. All-glass delivery apparatus was assembled for common solutions to minimize concentration changes. All analytical equipment assembled in one place for the convenience of the analysts.

In addition to standard methods already in use, other methods were investigated. Volumetric tubalioy methods were checked and the silver reductor methods found satisfactory. For the present, however, the standard gravimetric methods have been retained as most convenient for routine analysis.

Micro methods for tubalioy were investigated. No present methods are satisfactory for our purposes. The polarograph method is being studied as a

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possible solution to the problem. Colorimetric methods will be used for some purposes. Precipitate volume methods show some promise as a result of preliminary experiments.

Routine activities consisted of quantitative and qualitative analysis on 31 samples from production and chemical groups.

Research work that is in progress or planned consist of the following investigations:

1. Polarographic analysis
2. Spectrophotometric methods for tuballoy
3. Precipitate volume methods for tuballoy
4. Micro-gravimetric analysis.

Submitted by J. M. Carter (Part I)

M. D. Kamen (Part II)

H.E. Larson (Part III)

Information Division
1-8-46
ew

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Apparatus for Preparation of $TiBr_4$

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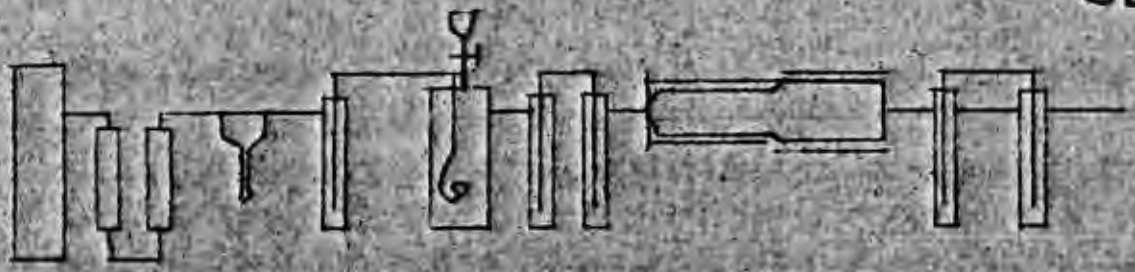
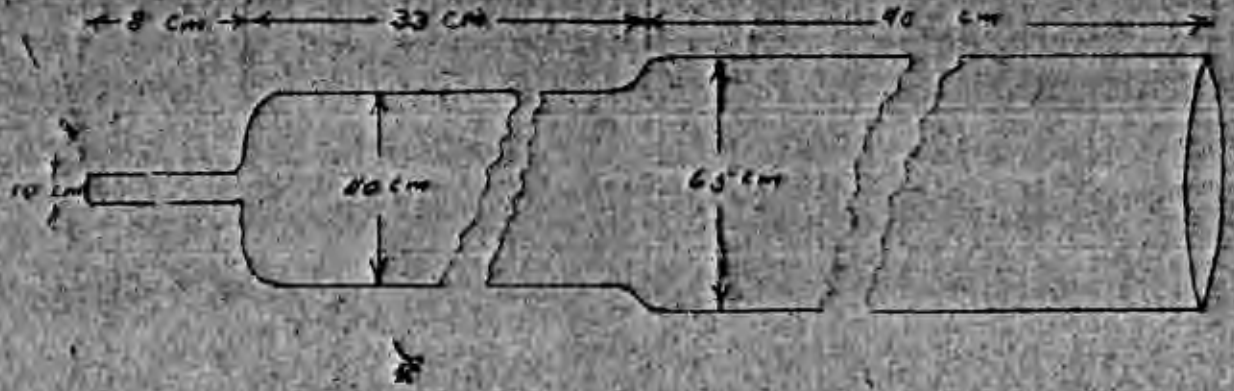


A N_2 tank
B $CaCl_2$ drying tube
C P_2O_5 drying tube
D Flow meter
E Trap
F Br_2 container
G Trap
H Conc. H_2SO_4 drying tube
I Quartz tube and oven
J Trap
K NaOH soln. to remove Br_2

Quartz Tube



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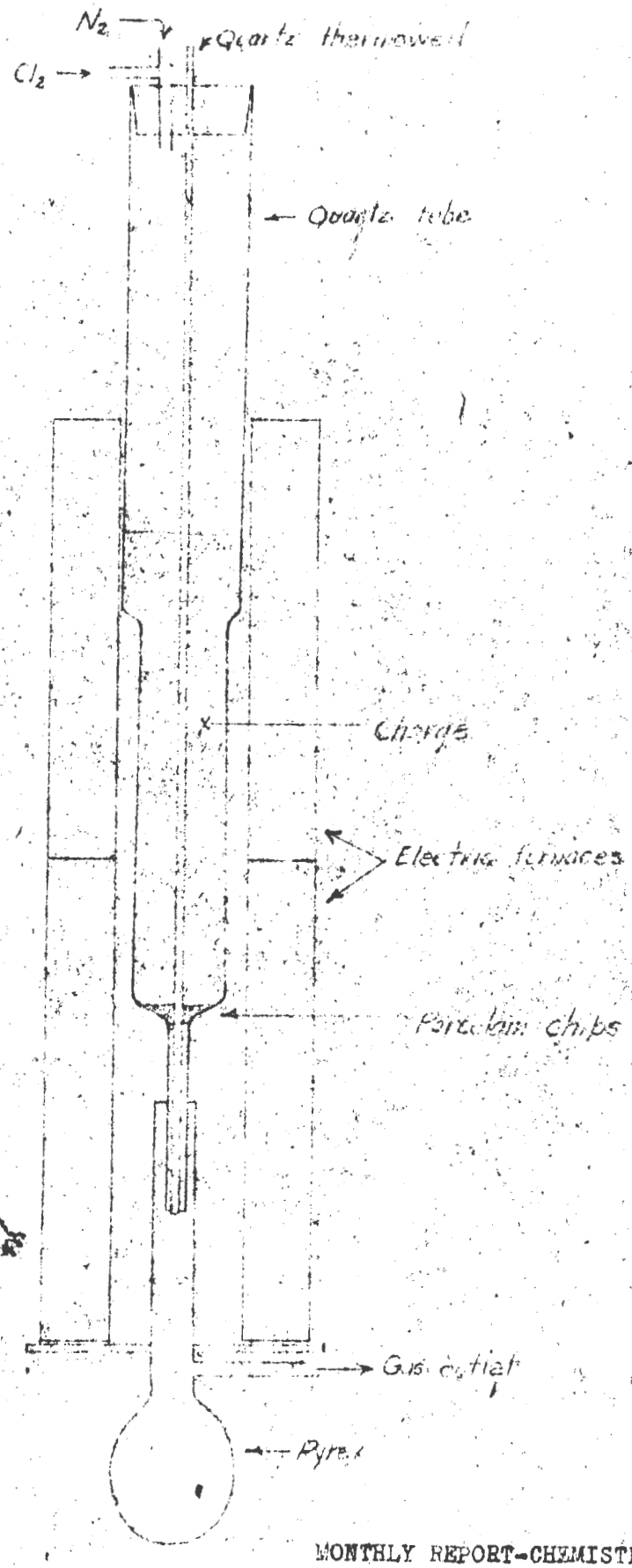

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Apparatus for prep. of $TeCl_4$

For the reaction using S_2Cl_2, the only changes were substitution of SCl_2 for CCl_4 in the dropping funnel, and Cl_2 for N_2 in the gas inlet

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Improved Vacuum Still for T City

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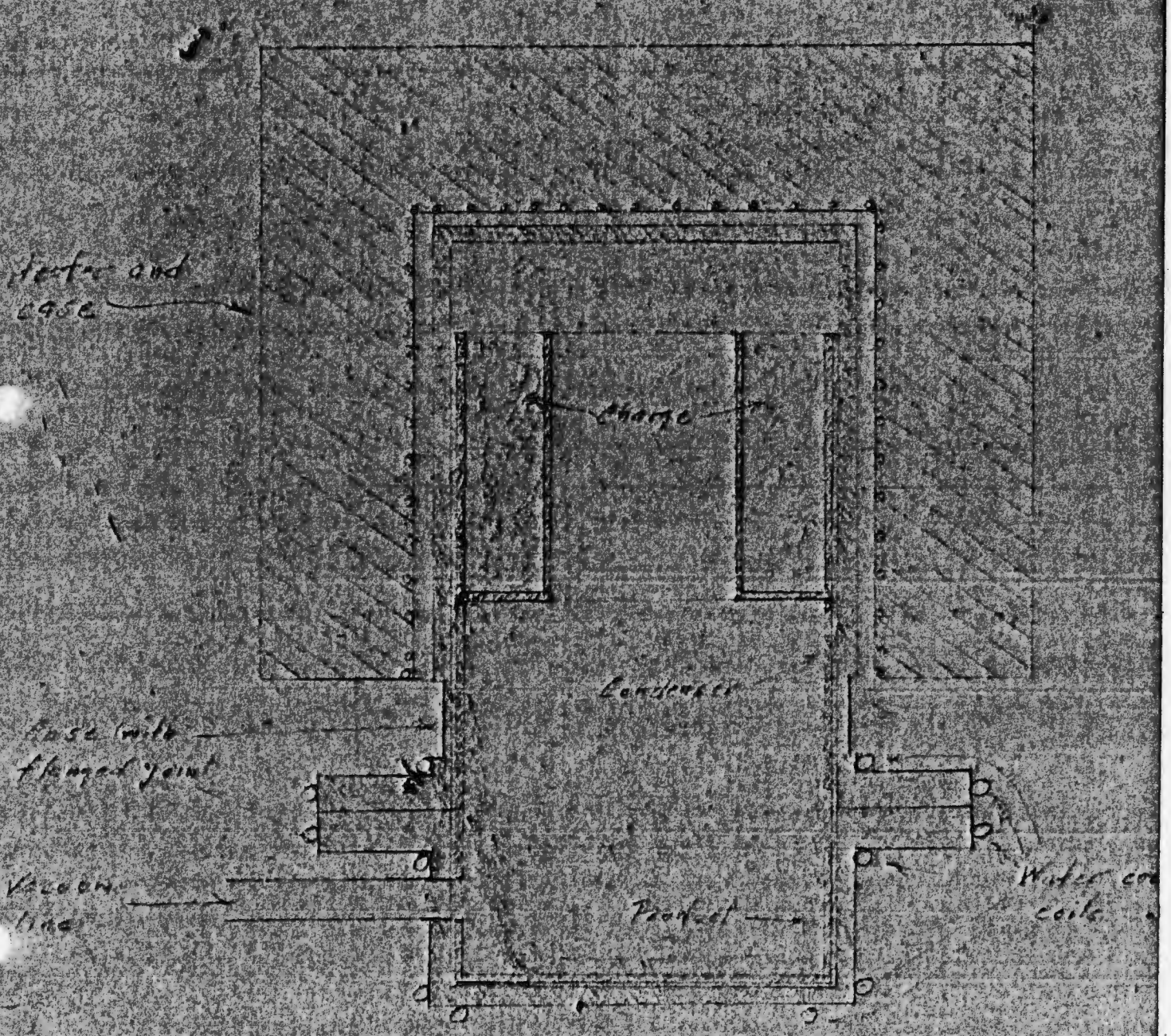

Fig. 6.

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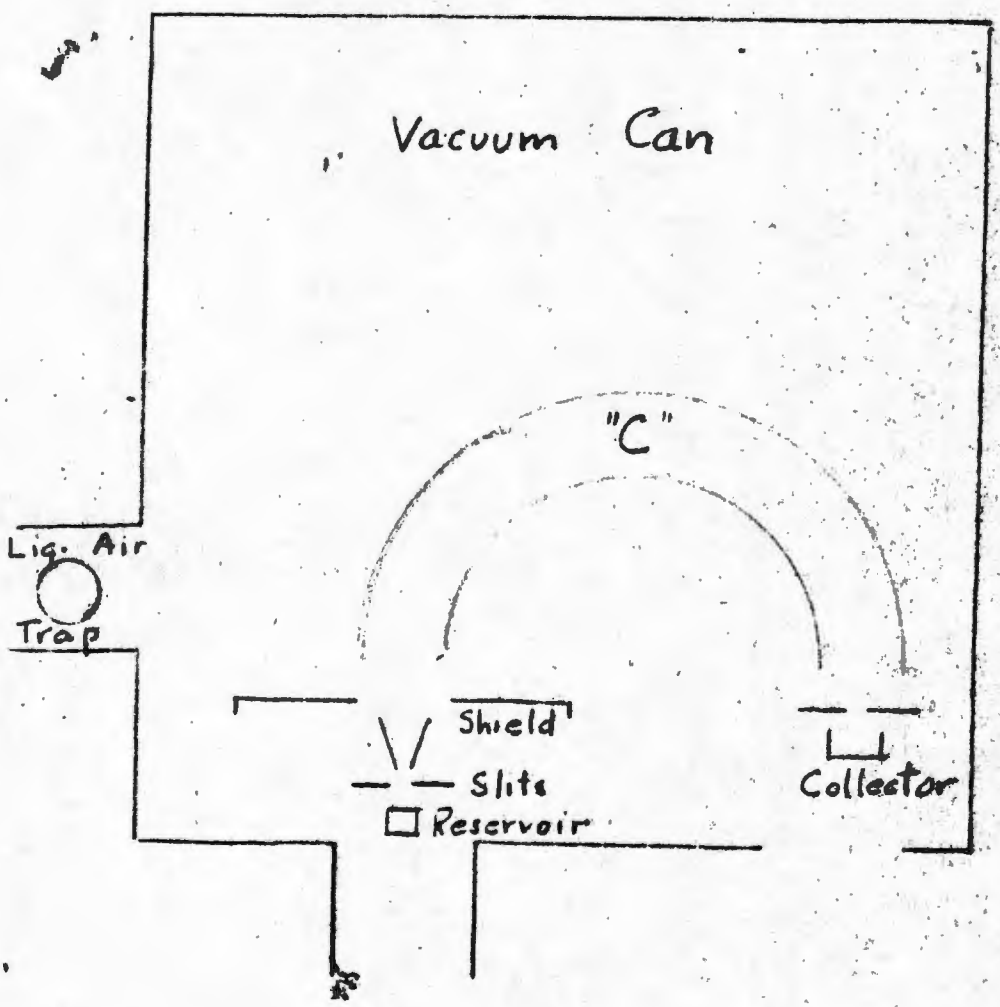

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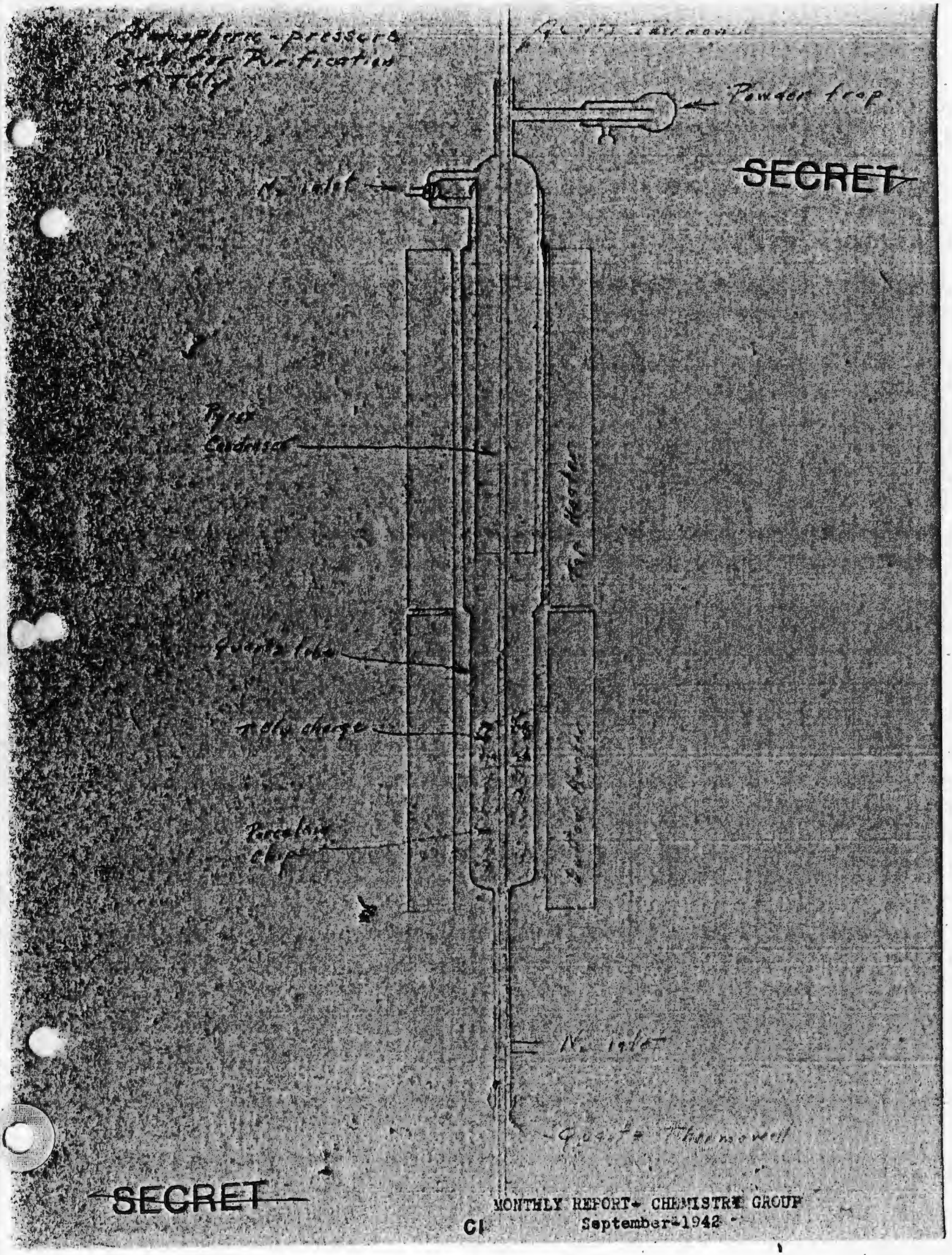
Powder trap
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Pyrex
Charge tube
Porcelain
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Department of Chemistry
BROWN UNIVERSITY
Providence 12, Rhode Island

Metcalf Research Laboratory
Charles A Kraus, Director

October 27, 1945

Captain John D. Ireland,
U. S. Engineer Office,
Box E,
Oak Ridge, Tennessee

Dear Captain Ireland:

In response to your wire, I am enclosing a brief account of the development of the work at Brown University in relation to the Y-12 project.

I might have written at greater length but I think that what I am sending on will give you a fairly good picture of the course of the developments and our relations to the Y-12 project.

If there is anything more than you want, please let me know.

Sincerely yours,

Charles A. Kraus

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BRIEF ACCOUNT OF THE DEVELOPMENT OF THE Y-12 PROJECT
AT THE METCALF RESEARCH LABORATORY, BROWN UNIVERSITY

The earliest work on the uranium problem was initiated at Brown University about January 1, 1942 under O.S.R.D. Contract OEMsr-290. This work was undertaken at the instance of Professor Harold C. Urey, of Columbia University, and was concerned with the problem of separating uranium isotopes by electrolytic means. This work was continued until about September 1, 1942 and much valuable information was accumulated with regard to the properties of several uranyl salts in aqueous solution. The work is comprehensively reported in Report No. BT-10, entitled "The Properties of Aqueous Solutions of Some Uranyl Salts", under date of October 26, 1942. Early in July, 1942, Dr. Kraus visited the Metallurgical Laboratory project in Chicago at the instance of Dr. A. H. Compton and, after studying the problems there, suggested that the Brown laboratory could usefully undertake a study of the reduction of uranium from its compounds to metal form by means of metals such as sodium and magnesium. An outline of the investigations and estimates of the cost were sent to Dr. Compton but the problem was not assigned. In the meantime, the problem was undertaken at Ames and at various other places, much along the lines originally suggested by Dr. Kraus.

About the middle of August, 1942, Dr. Rosen, acting in behalf of Mr. Murphree, suggested that the Brown Laboratory study the problem of producing metallic uranium by the reduction of uranium halides with sodium or other similar metals. This led to a second contract with the OSRD, OEMsr-688, for the purpose of studying this problem. Prior to that time, no work had been carried out looking toward the reduction

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of chlorides of uranium; earlier work had been directed toward the reduction of uranium tetrafluoride. Therefore, one of the first steps in the development of this problem was a study of means for producing uranium tetrachloride on a commercial scale. Experiments along these lines began early in September, being largely in the nature of exploratory investigations; first, by reaction of oxides with various chlorinating agents, such as CCl_4 and $SgCl_2$ in the vapor phase, and, later, chlorination of uranium oxides by means of sulfur monochloride and carbon tetrachloride in the liquid phase. (See Reports Nos. BT-6, BT-7, BT-11, BT-13, dated September 17, October 7, November 2 and November 17, 1942 respectively). The first considerable quantity of uranium tetrachloride was made by reaction of UO_3 and UO_2 with $SgCl_2$ in the liquid phase near the boiling point.

Samples of uranium tetrachloride were sent to Dr. Lawrence on September 28, 1942 and quantities of materials, ranging from one to ten pounds were shipped to Dr. Gilman, to Dr. Smith, to the Westinghouse Company and to Dr. W. S. Calcott, during November. The California group became interested in the methods employed at Brown for producing uranium tetrachloride and Doctors Carter and Boyer visited the Brown laboratory early in December, 1942. About this time, improved liquid phase reactors had been constructed and uranium tetrachloride was produced by the reaction of UO_3 and carbon tetrachloride in the liquid phase at temperatures up to 160°C. Between January 1 and June 1, the total of 262 lbs. of uranium tetrachloride was shipped to the Radiation Laboratory in California.

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Also in May, 1943, a complete reactor unit was shipped to the Radiation Laboratory and George C. DeGroot went to California to set up the reactor and demonstrate its operation. Such a setup was made at Davis and the reactor was operated there for some time.

Dr. McNally, of the Tennessee Eastman Corporation, first learned of the work at Brown University on a visit to the Radiation Laboratory early in 1943 and, on February 17, 1943, Dr. McNally, together with Mr. Ballard, Mr. Fellows and Dr. Geddes, visited the Brown laboratory and conferred at some length concerning the utilization of the liquid phase reaction with carbon tetrachloride for the production of uranium tetrachloride in the alpha process. As a result of this conference, the liquid phase reaction with carbon tetrachloride was adopted and put into practice at Y-12. In the meantime, work was carried out early in 1943 on the development of a small liquid phase reactor for the beta process. All details of this reactor were carefully worked out and the reaction was studied to determine the conditions under which to carry out the reaction, the precautions necessary to avoid losses and to cut down the time and to determine the yields, purity of materials, material balances, etc. This work is reported in Technical Report No.1 November 3, 1943. A reactor was sent to Y-12 on October 21, 1943. This type of reactor has been used exclusively in the beta process, at least up to July, 1945, and is still being used.

When the beta process, as originally laid out, came to be put in operation, great difficulty was experienced in the recovery of recycle material, since this process depended entirely on chemical methods of separation. It seemed necessary, therefore, to resort to an extraction process using ether as extraction solvent. Aside from the fact that the

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extraction efficiency of ether is not too favorable, the high volatility and low flash point of ether constitutes a serious hazard and, for that reason, work was undertaken at the Brown laboratory early in 1944 to find solvents less hazardous and more efficient than ordinary ether. For this purpose, the polyethers were developed. Samples of these ethers were synthesized for the Brown Laboratory by the Carbide and Carbon Chemicals Company and their properties as extraction solvents were investigated. Two ethers, dibutyl "Carbitol" and dibutoxytetraethylene glycol, were found to meet all the needs of the Y-12 project, both for production and for analytical purposes. Much of the work on extraction solvents was carried out subsequent to July 27, 1944, and the results appear in reports subsequent to that date.

As will be evident from the foregoing, the Brown laboratories were never requested to do the work which they actually did on the Y-12 project; this work was taken up by the Brown laboratories largely on their own initiative. It was only by chance that the Brown laboratories found an opportunity to study the problem of the reduction of the uranium halides and it was thus by the merest chance that the Brown laboratories had available toward the end of 1942 quantities of uranium tetrachloride and means for producing such chlorides by a practical process. Appended is a list of reports covering the period September 1, 1942, to July 27, 1944, which adequately substantiates any statements made above.

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COST ESTIMATE

The costs of the investigation at Brown, relating to the Y-12 project, cannot be determined precisely but they have been estimated rather reliably and the figures are given below for the period from ~~the period from~~ September 1, 1942 to July 27, 1944.

Salaries and Overhead	$73,000.00
Equipment and Materials	20,000.00
Total	$93,000.00

Charles A. Kraus

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REPORTS ISSUED FROM BROWN UNIVERSITY PRIOR TO JULY 27, 1944 WHICH CONTAIN INFORMATION RELATING TO THE PROJECT AT Y-12

1. TECHNICAL REPORTS

Brown U. File No.	Other Designation	Date Issued	Report Title	Remarks
BT-6	---	9/17/42	Monthly Report	First reference to investigations on preparation of Uranium Chloride
BT-7	---	10/7/42	Uranium Tetrachloride-Preliminary Report	Use of CCl_4 and S_2Cl_2 in the Vapor Phase. Liquid phase reaction with S_2Cl_2
BT-11	---	11/2/42	Progress Report	Liquid Phase CCl_4 and S_2Cl_2 reactions. First mention of Autoclave reaction
BT-13	---	11/17/42	Progress Report-Preparation of RCl_3, RCl_4 and RCl_5	Discusses results with a larger reactor
BT-14	---	12/1/42	Progress Report	Large scale preparation of UCl_5 by Liquid Phase CCl_4 reaction discussed; also S_2Cl_2 reaction
BT-15	---	12/15/42	Progress Report	Discussion of Liquid Phase CCl_4 reaction
BT-16	---	1/1/43	Progress Report	" " "
BT-17	---	1/9/43	Progress Report	" " "
BT-18	---	2/1/43	Progress Report	" " "
BT-19	---	3/1/43	Progress Report	Production of UCl_6 also

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1. TECHNICAL REPORTS - Continued

Bruce U. File No.	Other Designation	Date Issued	Report Title	Remarks
BT-20	---	4/1/43	Progress Report	Production of UCl_5 and UCl_6 discussed
BT-21	---	4/15/43	" "	"
BT-22	---	6/1/43	Summarizing Report of Investigations Relating to Uranium Chemistry Covering the Period Sept. 1, 1942 to April 15, 1943.	
BT-23	---	7/1/43	Progress Report	Production of UCl_5 and UCl_6 discussed
BT-24	A-1085	8/1/43	" "	" "
BT-25	A-1086	9/1/43	" "	Production of UCl_5 and UCl_6 discussed. Production of UCl_4 in liquid phase discussed.
BT-26	A-1087	10/1/43	" "	Production of UCl_4 in liquid phase discussed.
BT-27	A-1089	11/1/43	" "	Corrosion studies and use of the liquid phase process for conversion of oxide to chloride in the B-Process
BT-28	A-1088	11/3/43	Technical Report No. 1 A study of the production of UCl_4 by the reaction of UO_3 and CCl_4 in the Liquid Phase	
BT-29	A-1090	12/6/43	Progress Report	Studies on the Production of UCl_6
BT-30	A-1091 SC-1519	2/22/44	"A" Report - Technical Report Covering the Results of Investigations Carried Out During Dec., 1943	

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1. TECHNICAL REPORTS - CONTINUED

Brown U. File No.	Other Designation	Date Issued	Report Title	Remarks
BT-31	A-1092 CC-1685	3/10/44	"A" Report - Technical Report Covering the Results of Investigations Carried Out During Jan., 1944	
BT-32	A-1093	4/28/44	"A" Report - Technical Report for the Month of February, 1944	
BT-34	A-1095 CC-1716	7/7/44	"A" Report - Technical Report for the Month of March, 1944	
BT-35	A-1096 CC-1717	7/29/44	"A" Report - Technical Report for the Months of April, May and June, 1944	

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The last few of the above mentioned reports contain information on various miscellaneous subjects, such as the preparation of NF_4, reaction of various chlorides with anhydrous HF and studies on oxide.

2. "M" or MEMORANDA REPORTS

Brown U. File No.	Date Issued	Title
M-3	1/20/44	Preparation of MCl_5 by reaction of Active MO_3 (Eastman) and CCl_4 in the Liquid Phase
M-4	1/31/44	The Determination of M^{IV} as a Method of Determining the MCl_4 Content of MCl_4 - MCl_5 Mixtures
M-5	2/1/44	The Vapor Pressure and Freezing Points of CCl_4 - Cl_2 Mixtures

Brown U. File No.	Date Issued	Title
BM-7	4/19/44	Thermal Decomposition of Hydrated HO_4
BM-8	5/24/44	Investigations on the Distribution of HO_2 $(HO_3)2$ Between H_2O and Organic Solvents
BM-9	5/29/44	Investigations on the Distribution of $HO_2(HO_3)2$ Between H_2O and Organic Solvents
BM-10	6/6/44	Investigations on the Distribution of $HO_2(HO_3)2$ Between H_2O and Organic Solvents
BM-11	6/15/44	Preparation of Calcium Fluoride Beats
BM-12	6/28/44	Investigations on the Distribution of $HO_2(HO_3)_2$ Between Organic Solvents and H_2O
BM-13	7/1/44	Investigations on Gunk Solution
BM-14	7/16/44	Corrosion of Metals by Various Solutions

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• This is the first report issued relating to the extraction problem and the use of polyethers as extraction agents.

BM-13 Supplement No. 1	7/6/44	Corrosion of Metals by Various Solutions
BM-14	7/1/44	Distribution of $HO_2(HO_3)_2$ Between Organic Solvents and H_2O - System Dibutoxytetraethylene Glycol-H_2O

3. "P" or Progress Reports (written particularly for the (U. S. Engineer)

Date Issued	Title
2/18/44	Special Report - Outline of Investigations Program Carried out at Brown University under Contract W-7405-eng-73

Date Issued	Title
2/28/44	"F" Report for the Month of February, 1944
	3. "F" or Progress Reports (written particularly for the U.S. Engineer) - Cont'd.
4/26/44	"F" Report for the Month of April, 1944
5/29/44	" " " May, 1944
6/28/44	" " " June, 1944
7/27/44	Special Report - Outline of the Investigational Program as Presently Under Way and Contemplated at Brown University Under Contract W-7405-eng-75 and Extension Thereof

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April 1, 1943
Letter Contract No. W-7405
eng-48
New York, New York

University of California
Berkeley, California

Att: Mr. Robert M. Underhill

Gentlemen:

In contemplation of the early negotiation of a contract with you, which contract will be effective as of April 1, 1943, whereby you will engage in research and development work for this District of the same nature as you are now conducting at the University of California not covered by Contract No. W-7405-eng-36, the United States of America, acting through the undersigned Contracting Officer, hereby places an order with you that you shall, in the shortest possible time, furnish the labor, materials, tools and equipment, and services, and do all things necessary to conduct such research and development work as shall be requested in writing by the Contracting Officer or his duly authorized representative.

Funds for carrying out this work have been appropriated and are now available for the use of the War Department under Procurement Authority No. NRS 31110 P&S-07 A-0905-23.

The Secretary of War finds that it is in the interest of the War effort that this work be not delayed awaiting the negotiation of a formal contract.

It is contemplated that this contract will be supplemented by a more formal contract between yourself and the United States of America. Such supplemental contract will include an appropriate clause providing for the termination thereof for the convenience of the Government. All applicable contract clauses required by Federal Laws and Executive Orders to be incorporated in such formal contract are hereby incorporated herein by reference and will be incorporated in the formal contract and in all subcontracts hereunder. Pending the execution of such formal contract, reimbursements shall be made to you for such items of actual cost incurred by you as are approved or ratified in writing by the Contracting Officer or his duly authorized representative; provided, however, that in no event shall such reimbursements exceed the limitation on obligations set forth in the next to last paragraph of this letter contract.

Upon your acceptance hereof advance payments in accordance with the existing requirements of the War Department may be made to you upon your application.

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Neither this contract, nor any interest therein, nor claims thereunder shall be assigned or transferred by you to any other party or parties.

It is understood that disclosure of information relating to the work contracted for hereunder to any person not entitled to receive it, or failure to safeguard all secret, confidential and restricted matter that may come to you or any person under your control in connection with the work under this contract, may subject you, your agents, employees, and subcontractor to criminal liability under the laws of the United States. (See Title I of an Act approved June 15, 1917, 40 Stat. 217; 50 U.S.C. 30-42), as amended by an Act approved March 28, 1940 (54 Stat. Chap. 72); and the provisions of an Act approved January 12, 1938 (52 Stat. 3; 50 U.S.C., Supp. V 45-454), as supplemented by Executive Order No. 8381, dated March 22, 1940, 5 F. R. 1147, D. I. You shall cause a like provision to be inserted in all subcontracts under this contract.

In the event that the United States of America is unable to negotiate with you a satisfactory contract on or before June 1, 1943, this contract will terminate and the United States of America will pay you in full settlement thereof a sum equal to reimbursement for all costs incurred by you in connection with the performance of this contract plus such other sums as have actually been expended by you in good faith in settlement of all obligations, commitments and claims which you may theretofore have incurred, less any reimbursements previously made, but in any event the total payment shall not exceed the sum of One Million Dollars ($1,000,000.00).

If the foregoing is acceptable to you, it is desired that you so indicate hereon and on the enclosed two copies of this letter contract and return the original and one copy to the Contracting Officer. Such acceptance will constitute your notice to proceed.

Very truly yours,

THE UNITED STATES OF AMERICA

BY /s/ J. C. Marshall
J. C. Marshall
Col., Corps of Engineers
Contracting Officer

Accepted the date 7 day of April 1943

UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA

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WAR DEPARTMENT
UNITED STATES ENGINEER OFFICE
Madison Square Area
P. O. Box 42
Station F
New York, New York

NYSM 0-243-a1 MB

29 November 1944

Dr. R. T. McBee,
Purdue University,
Lafayette, Indiana.

Dear Dr. McBee:

This office has been informed by Lt. Colonel Ruhoff that the manpower and facilities at present available at Purdue University are urgently needed by his office in connection with certain research work desired by him.

Accordingly, this is to inform you that this office wishes to cooperate with Lt. Colonel Ruhoff to expedite arrangements for him to utilize the capacities of your research group. Lt. Colonel Ruhoff has been informed that during the period 1 December 1944 to 1 January 1945, you will be requested to arrange your work so that personnel will gradually be shifted from NDA work to work for his office, and that by 1 January 1945, your entire staff will be engaged in work for his office.

This office feels that the latest discovery at Purdue of an azeotropic distillation process capable of separating 716 in a high state of purity from the more volatile fractions of crude 71501, has brought the laboratory phases of the 71501 process to a high state of perfection. It appears to this office that most of the remaining work on this process can best be carried out on the semi-works scale in those plants where the process would actually be used.

Therefore, you are requested to transfer your personnel to Lt. Colonel Ruhoff's project beginning 1 December 1944. This transfer should be carried out as rapidly as is consistent with the orderly completion of those phases of the laboratory work on the 71501 process which appear to you to be most in need of a small amount of further experimentation. The transfer should be complete by 1 January 1945, so that by that date, none of the research staff at Purdue will be working on projects for the Madison Square Area.

It is understood that Contract W-7405-eng-74 will be administered by this office until 1 January 1945. After that date, it is understood that Lt. Colonel Ruhoff's office expects to assume the

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administration of this contract, and that a new supplement to the contract will be prepared for continuation of the work after the first of January. Although the contract with the Purdue Research Foundation will be under the administration of Lt. Colonel Ruhoff's office after that date, we are hoping to be able to count on having the benefit of your advice, on a consultant basis, after 1 January 1945.

Very truly yours,

W. E. KELLY,
Major, Corps of Engineers
Area Engineer

cc: Lt. Col. Ruhoff

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B. Burford, III

December 12, 1944

Captain W. M. Hearon
War Dept. - U. S. Engineer Office
Madison Square Area
P. O. Box 42, Station F
New York 16, New York

Dear Captain Hearon:

Confirming a recent telephone conversation between this office and Major Thompson of your office, we wish to submit the following amendment to our Scope of Work under this contract.

Pursuant to the request of Lt. Col Ruhoff for certain work to be done here by a group of persons not to exceed 15 in number, we submit for your approval the following: We propose to undertake research on the recovery of the Tube Alloy from process materials, and conduct X-ray investigations, as directed by Col. Ruhoff. This is in addition to the present work being conducted for your office, and will utilize a staff composed principally of chemists whose backgrounds have been in physical, inorganic, and analytical chemistry.

It is estimated this work for Colonel Ruhoff will require the expenditure of $10,000 per month, while the program being carried out for your office will require the expenditure of $15,000 per month. Therefore, to carry both phases of the work to February 15, 1945, we will require an additional appropriation of $37,500. It is believed that the present appropriation will very nearly equal our expenditures up to and including December 31, 1944.

Hoping you find the above satisfactory, and awaiting your authorization to proceed with the work as outlined, I am with best personal regards,

Very truly yours,

W. B. BURFORD, III

CC: Lt. Col. J. R. Ruhoff

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PURDUE UNIVERSITY
Department of
Chemistry
Lafayette, Indiana

January 23, 1945

Major G. W. Russell
United States Engineer Office
Manhattan District
Oak Ridge, Tennessee

Re: Scope of Work
[redacted]-133-a
W-7405-eng-74

Dear Major Russell:

Purdue University and the Purdue Research Foundation acknowledges your letter of January 16, 1945, canceling all previous scopes of work agreements which the Foundation has had with the U. S. Engineering Office Under Contract W-7405-eng-74.

The following scope of work for Supplement No. 5 of contract W-7405-eng-74, effective January 1, 1945, is submitted for approval.

(1) Research and development in connection with the recovery of U from process materials and the conversion of U into a compound suitable for process uses.

(2) Research in the development of the X-ray spectrograph as a method for the determination of U.

(3) Research and routine test work for the determination of hydrogen in fluorine-containing organic compounds.

Approval of these three items, effective January 1, 1945, as the scope of work for the Foundation under Supplement No. 5 of contract W-7405-eng-74 is requested.

Very truly yours,

H. T. McBee, Professor
Organic Chemistry

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ARMY SERVICE FORCES
United States Engineer Office
Manhattan District
Oak Ridge, Tennessee

[redacted]-33-b

31 January 1945.

Professor E. V. McBee,
Department of Chemistry,
Purdue University,
Lafayette, Indiana.

Dear Professor McBee:

Receipt of your letter of 25 January is acknowledged. Scope of work as stated in this letter is approved effective as of 1 January 1945.

Item number 5 in the above scope of work will be formally cancelled when the transfer of the hydrogen determination apparatus is completed in accordance with recent requests.

For the District Engineer:

Very truly yours,

G. W. Russell,
Major, Corps of Engineers,
Assistant.

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WAR DEPARTMENT
United States Engineer Office
Madison Square Area
P. O. Box 42
Station F
New York 16, New York

KHM 0-243-el MS

16 December 1944.

Dr. W. S. Durford,
P. O. Box 35,
Waverly Station,
Baltimore 18, Maryland.

Dear Dr. Durford:

Reference is made to your letter of 12 December submitting an amendment to the scope of work under Contract W-7401-eng-43. The scope of work as submitted in your letter is hereby approved. A supplement No. 8 is being prepared to the contract extending the expiration date to 15 February 1945, and increasing the appropriation by $37,500 to provide for carrying out the work for Lt. Colonel Ruhoff and for the New York Area. After 15 February 1945, it is expected that administration of this contract will be taken over by Lt. Colonel Ruhoff.

Very truly yours,

K. D. KELLEX,
Major, Corps of Engineers
Area Engineer.

Lt. Col. J. R. Ruhoff.

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CHEMISTRY OF THE POLATRON PROCESS

J. M. Carter and M. D. Kamen

June 11, 1943

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GENERAL AND ALPHA PROCESS

J.M. Corbor

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M.D. Kamen

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GENERAL AND ALPHA PROCESS

J.M. Carter

Introduction

During a conference in Boston, the desirability of having a summarizing report of the chemical work on tuballoy was pointed out. This report has been written as a result, and deals only with the chemistry of tuballoy which is directly involved in the calutron process. There is much other chemistry of tuballoy which has been investigated in Berkeley, but this will not be covered here.

Briefly, the calutron process involves the following, which affect the chemical operations:

1. Preparation of volatile tuballoy compounds from oxides or recovered material. After a number of tests on various of these compounds, only the chlorides TCl_4 and TCl_5 have been found suitable.

2. Purification of the chlorides, and loading into the proper evaporation bottles for use in the calutron.

3. Evaporation of the chlorides in the calutron, whereby 5 to 8 per cent is ionized and 3 to 5 per cent used. The rest of the material is redeposited in an essentially unchanged form throughout various parts of the equipment. The ionized material which is not used is found mostly as metal mixed with sputtered material from the equipment.

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4. The purification of the recovered materials from Process 3, to remove the substances introduced from the equipment.

5. The conversion of the purified tuballoy to oxides for use in Step 1.

6. The recovery of the usefully ionized material in two separate lots, one of which comprises the product, the other being spent material to be rejected.

There are two principal sources of inefficiency in the calutron process. The first is that, as mentioned above, only 3 to 5 per cent of useful ions are produced. All the other 90 odd per cent of material must be recovered and recycled in order to get all the product out of the incoming charge. The second and more important of the inefficiencies is that complete separation of the wanted material from the bulk of the charge is not accomplished in one process, but requires two. On this account, the second process must, of necessity, operate on extremely valuable material which has been produced by the first process; hence it requires different techniques in handling and extraordinary precautions to avoid any losses. If either or both of these inefficiencies could be removed, the entire process would be much simpler and a great deal less expensive, and, in either case, the chemistry involved would be of rather insignificant proportions.

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Chemical Properties of Materials Involved.

The halides of tubalioy are, in general, typical non-polar, inorganic compounds, similar to aluminum chloride, silicon bromides, etc. Because of their hygroscopic and corrosive properties, they must be prepared, stored and handled under anhydrous conditions, and frequently out of contact with air. Some early work on suitable materials involved tubalioy hexafluoride, tubalioy tetrabromide and tetraiodide, and some fairly complex organic materials containing tubalioy. A comprehensive survey showed that the tetrachloride, TCl_4, was the most suitable for use in the polatron process. Subsequent discovery and preparation of the hexachloride indicated that this might also be satisfactory, but, for the initial operation, the use of the tetrachloride has been decided on. TCl_4 is stable under ordinary conditions when dry. It has a fair volatility ($p = 1/10$ mm. at 450°C) and is not extremely corrosive. In addition, it is the most easily made and purified of all the halides. The possible exceptions are TF_4 and TF_6, which are easily made and purified but are otherwise non-suitable.

In connection with work on TCl_4 made by a method developed by Professor Kraus of Brown University, it was found that this material could be decomposed in a vacuum to TCl_4 and the hitherto unknown hexachloride, TCl_6. This material is much more volatile ($p_{TCl_6} = 0.1$ at approximately 80°C) and hence offers advantages for use in the polatron process.

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However, it is rather unstable, losing chlorine to give TCl_3 and TCl_4 at temperatures near 100°C. Also, this decomposition appears to be accelerated by various metals and graphite. It reacts much more rapidly with metals than TCl_4 does, resulting in both decomposition of the TCl_5 and corrosion of the metal. Possibly the greatest disadvantage of the TCl_5 is its great reactivity with water. Hydrolysis and breakdown to lower chlorides occur with concentration of water vapor as low as 0.03 mm. Hg. This necessitates the greatest care in handling the material. Up to the present the only satisfactory method of handling it bottles has been to fill and seal them off under vacuum.

The chemistry of tuballoy as it enters into the purification and recovery processes is somewhat complicated, but not difficult. In aqueous solutions two valences occur, the $+4$ and the $+6$, and in general the properties are those which would be expected. The element which most closely resembles it is iron, but there is enough of a difference so that separation from iron impurities is not difficult.

A series of reports on the chemistry of tuballoy in aqueous solution, principally in connection with methods of analysis, has been prepared, largely under the supervision of Dr. Larson. Other work of this nature is reported in more detail in the following section of this report, by Dr. Kamen.

In connection with the use of the TCl_4 in the palatron process, the question of corrosion of equipment arises, as

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it affects the durability and also the purification of the recovered tuballoy. There are several kinds of corrosion which are encountered. First, there is corrosion by the hot TCl_4 vapor at approximately 300^0C. This type of corrosion arises from two different properties. The material is rather easily reduced to the substance TCl_4, giving off an atom of chlorine. The chlorine then behaves as would be expected at this temperature, attacking a number of metals. In addition, the tetrachloride vapor seems to have a corrosive action of its own, as such, and combines with a number of metals to form compounds of indefinite composition but which definitely corrode the material. The second type of corrosion which is encountered is that due to the action of water on the tetrachloride in removing it from the equipment. As soon as water is added, the tetrachloride hydrolyses rather completely, giving fairly strong solutions of hydrochloric acid. It is unfortunate that such materials do not withstand the corrosion by gaseous chlorine and by hydrochloric acid in water. A third type of corrosion is that due to the sputtering action of the ions, which are formed in the equipment, on all the parts with which they come in contact, and here again the materials which withstand the sputtering the best are not necessarily those which will withstand the chemical corrosion. This problem of corrosion is covered in a series of reports, largely by Mr. Cummings.

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For withstanding the action of the hot tetrachloride vapor the best materials are: gold, nickel, carbon, stainless steel, and a few minor metals which cannot be used for other reasons. Copper, silver, aluminum and iron seem to be particularly unsuitable, as they are attacked more or less rapidly by the hot vapor. On the addition of water to dissolve off the tetrachloride, most of the metals in the above list are fairly satisfactory, and about the same ones are badly attacked. Against the sputtered ions, the stainless steels and graphite are the best, while nickel is not very satisfactory. Metals with a poor sputtering characteristic at low pressures are especially bad (i. e. aluminum). In connection with the sputtering problem, there is an allied one, that of heat dissipation, because, in general, the ions carry a great deal of energy. It is on this account that stainless steel, which is otherwise extremely suitable, must be used with care where sputtered ions are encountered, because of its extremely poor heat conductivity.

General Flow Sheet.

The present setup provides for some thousand J's or converters in five hundred tanks for the alpha process and 144 J's in the seventy-two tanks for the beta process. It is possible that only about half of these latter will actually be required. All the above are to be operated on TCl_4. On the basis of the present operation, the TCl_4 required for the Alpha process

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will be about five hundred kilograms a day, of which somewhat less than 10 per cent will be produced from new materials from the outside and 90 odd percent must be made from material recovered from the equipment. This recovered material will be derived from approximately seventy tanks opened up every day. That is on the basis of five hundred tanks run on the seven-day cycle. At present, attempts are under way to run on a fourteen or even a thirty-day cycle. This will result, if successful, in only thirty-five or fifteen tanks opened per day. The product from the alpha process will amount to some three hundred grams a day, which must be recovered from about 140 receivers. This means that only about two grams (of enhanced tribality) will be present in each receiver, spread over an area more than a foot square. Longer cycles will give correspondingly larger amounts from each receiver. This product from the alpha process constitutes the feed for the beta process, but the amount required to operate the beta process is approximately sixteen to twenty kilograms, of which one half is handled each day. It is obvious then that the alpha process must run for some time at full capacity before the beta process can be operated in more than a very small way. In order to start the beta process just as rapidly as possible and to avoid having large amounts of valuable material tied up in it, the equipment on the beta process is designed to operate on a one-day cycle, and it is felt that the chemical recovery can also be held down to this time. The product from the

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beta process amounts to about fifty grams a day, to be recovered from sixty receivers.

Chemical Requirements of the Alpha and Beta Processes

These requirements are very different, as outlined below. In the alpha process the main objective is to produce the largest possible amounts of product. This product must not be lost or contaminated with lower grade material. Incomplete removal of recoverable material from the tanks, however, or losses in purification or in the re-formation of TCl_4 are not important, as new material to be fed into the process is relatively abundant and not overly expensive.

In the beta process the output is obviously limited by the amount of alpha product which is available for feed. Here there must be only a negligible loss of either product or the recycled material, and, further, all operations must be done as rapidly as possible, because of the large amounts of circulating material which are necessarily required at best. As mentioned before, there is also the problem of a very reduced output during the time required to build up the amount of material needed to operate the beta process. These questions are treated more in detail below.

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Specific Chemical Problems.

(a) The Preparation of TCl_4 - Prior to the work done on this project there were a number of methods of preparing TCl_4. None of them were extremely satisfactory. The most commonly used one involved the reaction

$$TO_2 + 2Cl_2 \rightarrow TCl_4 + CO_2.$$

This reaction proceeds only at temperatures of 500°C or above and obviously requires fused quartz equipment. In addition, it has been proven very difficult to get a satisfactory mixture of the tuballoy oxide and the carbon, and, in fact, the only method which has proven at all satisfactory is to mix the oxide with sugar and then to char the sugar to carbon. We have made a considerable amount of TCl_4 by this process, but it was early thought that there must be more convenient methods. The second method which we tried and which had been in use at the Bureau of Standards was the reaction

$$TO_2 + 2S_2Cl \rightarrow TCl_4 + 2O_2 + 3S.$$

This reaction requires about the same temperature as the preceding one and must also be carried out in quartz. There is not the problem of mixing the oxide with carbon, but, on the other hand, there is a great disadvantage in that the product always contained sulfur, and, in view of the ever-present leaks in such apparatus, the sulfur gases were hard on the operators. After investigating the above, and a number of other reactions which did not prove suitable, it was finally found that tuballoy dioxide, as well as the

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other oxides, could be reacted with carbon tetrachloride vapor at temperatures as low as 390°C, according to the reaction

$$TO_2 + 2CCl_4 \rightarrow TCl_4 + 2COCl_2.$$

Because of the cleanness and the suitability of this method, it was further developed at Berkeley and at Davis, and at present we have equipment which will turn out on the order of three pounds an hour of TCl_4 of about 99 per cent purity. The advantages of this reaction are the low temperature at which it can be carried out, (At present we operate at approximately 450°C.), and the purity and satisfactory physical nature of the product. There is also the flexibility as to the charge material, in that we have found that we can use TO_2, TO_3 and T_3O_8, as well as various oxychlorides and still residues. The disadvantages are that it so far has worked only in rather small units, although it is believed that this could be overcome; that, further, in the most satisfactory form of equipment, a continuous rotating furnace, small amounts of impurities formed caused the rotating elements to stick at first. This has now been solved. Possibly the most serious disadvantage is the formation of appreciable amounts of TCl_4 as a very fine dust which is rather difficult to remove.

Independently of the work at Berkeley, Professor Kraus of Brown University developed a liquid phase reaction involving TO_3 and liquid carbon tetrachloride. This gives as a product the pentachloride rather than the tetrachloride.

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But it can be carried out at temperatures not above 175°C and obviously is capable of producing much larger amounts in a single piece of equipment. It has the disadvantage that the reaction is rather difficult to control, as a very active oxide is likely to react all at once, and, on the other hand, oxides prepared by certain methods do not appear to react at all. There is a further disadvantage in that, if the tetrachloride is to be used, the pentachloride which is formed in this reaction must be decomposed, and as yet this has not been done to give a tetrachloride with the proper physical characteristics.

Several minor methods were investigated at Berkeley, including the reaction of tuballoy carbide with chlorine. This in itself is a very satisfactory reaction, as it occurs at temperatures of 400°C or lower and gives a very pure product. However, in view of the difficulty of converting recycled material into the carbide, which involves electric furnaces and a good bit of loss, this method was not investigated further. The second reaction, which is fairly suitable, is that of any of the oxides of tuballoy with liquid sulfur chloride. This proceeds in much the same way as the reactions with liquid carbon tetrachloride, but for some unknown reason is much less rapid and has the further disadvantage that very considerable amounts of sulfur are included in the product. Before use the sulfur must be removed, which is rather difficult to accomplish completely. A number of other chlorinating agents have

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been tried in connection with all the oxides of tubalioy. None are at all satisfactory. Most of them, in fact, fail to react at all, even those which it was felt would be the most reactive. There is one possibility which is especially interesting- avoiding the formation of TCl_4 in the gas phase reaction by the use of chloroform, either alone or in connection with carbon tetrachloride. The chloroform seems to have the effect of reducing very appreciably the amount of tubaliq/pentachloride, but there is the rather bad disadvantage that the chloroform itself tends to decompose with the formation of large amounts of carbon.

(b) Purification of TCl_4 and loading. Up to the present, practically all the TCl_4 which has been used in the calutron process has been distilled(sublimed) to free it from small amounts of solid impurities and from adsorbed gases and vapors. This process is carried out at about 650°C, under a vacuum of 10^{-4} mm. Hg. or better, in stainless steel stills developed at Berkeley. These stills produce approximately two kilograms of purified TCl_4 in about four hours.

Recently a large vacuum still, capable of handling sixty kilograms of TCl_4 in a six-hour run, has been constructed and tested, largely on the design of the Tennessee-Eastman Company. In general, this still operates quite satisfactorily, and, after some modifications required by the refractory nature of the material are incorporated, there should be no difficulty.

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It is still hoped and expected that, as more experience is accumulated, it will prove possible to use TCl_4 without distilling it. In view of the large number of other variables to be investigated in the pilotrun, it has not been thought advisable thus far to introduce a further variable of composition and physical state of the charge material. It is the opinion of the chemical group, however, that undistilled TCl_4 can be used, possibly requiring treatment to remove adsorbed gases and vapors.

The only rigid requirement on filling K bottles with TCl_4 is that the material be kept dry. This requires that the bottles be thoroughly dried and that they be filled in a dry atmosphere and adequately stoppered to prevent access of moisture.

As mentioned above, the use of TCl_4 requires much more complicated and careful treatment. The TCl_4 must be predared by decomposition of TCl_5. This is done at present in the same (small) stainless steel stills used for TCl_4, but heated to only some 200°C. This yields a sublimate of from 30 to 50 percent of the initial charge, having a TCl_4 content of 75 to 80 per cent. (The other 20 to 25 per cent is TCl_4 either as such or as TCl_5.) The sublimate is then redistilled and loaded into K bottles in an all-glass system developed in this laboratory.

(c) Recovery of Tuballoy from Pilotrun Equipment. As mentioned above, there are different objectives in the alpha and beta processes. In the alpha pro

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equipment ready for further use is the principal object. The material which is left on or in the equipment is of importance only insofar as it affects the subsequent operation. The total amount of tuballoy which is tied up in the alpha process is of minor importance. In the beta process, the equipment must of course be in shape for further use, but complete removal of tuballoy without any loss is a primary consideration. In addition, the tuballoy recovered should be in such a form so that losses in the subsequent concentration and purification will be held to a minimum. This requirement influences the types of material to be used in the beta process equipment. There are a number of steps in the recovery of the tuballoy from both of the processes. As most of the material is in the form of redeposited tetrachloride, a simple wash with water removes the greater part of it. Fortunately most of this is near the source and concentrated around the J block, so that the removal and washing of a small part of the equipment is enough to recover most of the material. Here again, the difference between the alpha and the beta operation becomes apparent. On the alpha equipment, as soon as parts are reasonably clean, any further rinses to remove the last small amounts can be discarded. On the beta process, however, even very minute amounts are extremely valuable, so that all the rinses must be saved and the tuballoy recovered from them. There is another approximately four to five per cent of the tuballoy that is deposited either as metal or as

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around in the equipment, nearer the receivers, and because of its nature this material must be recovered by the use of acids. These materials do not pick up water, they do not interfere appreciably with the operation of the equipment, and in the alpha process they can be allowed to accumulate for considerable periods of time. But, for the reasons given above, they must be removed from the beta equipment at much more frequent intervals. It is obvious that the tuballoy solutions recovered from the alpha process are much more concentrated than those from the beta, so that the liquid from the alpha can go directly into chemical purification. On the beta, because of the large amounts of water involved, the first step must consist of an evaporation, in order to build up the concentration of tuballoy to a point where it is convenient to work with it chemically. In addition, the material from the beta recovery is much more complex because of the acids which are used and also because of the variety of corrosion-resistant equipment which must be used in the beta process.

(d) Chemical Purification. In the alpha process, the principal impurity is copper, with minor amounts of iron and traces of other metals. In the beta process, because of the wide use of stainless steel and the almost entire avoidance of copper, the major impurity is iron, with the accompanying proportions of nickel and chromium ordinarily encountered in stainless. There are also the minor metals which occur in all cases and are due to such pieces of

equipment as filaments, soldered connections, anti-sputtering shields, etc. The methods of purification are rather simple and based on a number of well-known reactions of tuballoy. The first, which is well known and has been used for a long time, depends on the formation of a carbonate complex by tuballoy when it is in the oxidized state. By having both ammonium ion and carbonate ion present at the same time, iron and all other metals which form insoluble hydroxides not dissolving in excess ammonia can be precipated, leaving the tuballoy dissolved as the complex. This, together with a precipitation in which only ammonium is present, purifies the tuballoy from all elements likely to be encountered. The copper, which is the main contaminant in the alpha process, is removed as a dissolved copper ammonia complex when the tuballoy is precipitated by the use of ammonia without carbonate. A further method which was used by Professor Kraus largely as a means of preparing very reactive TO_3 has also been found useful here for purification. This depends on the precipitation of the compound TO_4 with hydrogen peroxide. This appears to be entirely specific for tuballoy, and the only element which interferes at all badly is iron, which, as is well known, catalyzes the decomposition of hydrogen peroxide. It has been found, however, that the iron can be sufficiently inactivated by forming a complex with such materials as lactic or acetic acids. A further method that has been developed largely in this laboratory is the controlled

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precipitation of tuballous hydroxide. This obviously requires a preliminary reduction, either chemically or electrolytically, and a rather careful control of all the conditions under which the precipitation is made. This method is discussed in greater detail in the subsequent section of this report by Dr. Hamm. A number of other methods have been proposed, involving the removal of copper by treatment with metallic iron, the formation of cyanide complexes for the removal of impurities, extraction of tuballay nitrate with ether, and a number of others, but the ones outlined above seem to serve the purpose well enough and are not as complicated as a number of others which have been proposed.

(c) Preparation of Oxide. The oxide that is to be made varies to a certain extent with the method that is chosen for the preparation of chloride. In the gas phase reaction with carbon tetrachloride the oxide TO_3 is by far the most desirable, in that the reaction is much more rapid than with other oxides and the temperature at which the reaction occurs is very convenient, both for obtaining TCl_4 of the proper physical characteristics and for the removal of such impurities as iron. Furthermore, less TCl_5 is formed. It is obvious that in this reaction, iron if not present in large amounts, causes no difficulty because any iron present will be converted to ferric chloride, which boils out of the apparatus at a conveniently low temperature. The liquid phase with carbon tetrachloride requires TO_3 as the

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starting material, as UO_3 does not appear to react. Furthermore, UO_3 from different sources reacts very differently. The most active material is that produced by the decomposition of UO_4 at 325°C in a partial vacuum. In small amounts it is very satisfactory, but in lots of three hundred grams or more, the reaction tends to go out of control and become almost explosive. Some forms of UO_3 do not appear to react at all. That prepared by decomposition of the nitrate reacts fairly satisfactorily, but each batch must be tested to determine the proper conditions.

(f) Recovery of Product. Elimination of Depleted Material. Both the product and the depleted material are essentially metal, contaminated by sputtered material from the receiver pockets and other parts of the equipment. There are likely to be considerable amounts of carbon and graphite, which introduce no difficulties into the recovery. The present design of the receivers calls for the use of stainless steel and graphite. The metal is rather tightly bound to the walls of the receiver and must be removed chemically. The depleted material is of essentially the same nature as the product, but, as the only requirement is that it be thrown out of the system, there is no particular problem on getting it out of the receivers. In fact, it may be allowed to accumulate until the receivers are essentially full and then be thrown out, together with the discarded receivers. In the beta process, this depleted material cannot be rejected until an analysis has shown that it is of

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as low grade as the incoming material which is used in the alpha process. The method of removing the product from the receivers is to treat the receiver material with a dilute solution of hydrochloric acid and hydrogen peroxide, which attacks tubalioy very vigorously but has relatively little effect on stainless steel and of course none at all upon graphite. The methods used for purification are in general the same as those employed for the recycled material.

Alpha Process and Equipment.

The methods and equipment for the alpha process are fairly well decided on at present, in marked contrast to the beta. A large part of the equipment is ordered and either completed or in process of fabrication.

For the preparation of the TCl_4, both gas phase and liquid phase equipment have been provided. This has been done, both as insurance to provide against unexpected difficulties in the operation of one or the other and because, at the time the equipment was ordered, the final decision as to whether to use TCl_4 or TCl_6 had not been made. Since the TCl_6 requires previous preparation of TCl_6, approximately double the capacity is required. The gas phase equipment provided consists of twelve continuous rotary furnaces, each expected to produce some seventy-five pounds of TCl_4 per day. In addition, two large (1,000 gal.) liquid phase reactors are on order. These reactors should produce about one thousand pounds of TCl_6 with each charge. The TCl_6 can be converted to TCl_4 either in the same reactors or in separate containers made for that purpose.

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The still equipment at present calls for four stills, each capable of taking 125 pounds at each charge. It is estimated that three charges per day can be run in each still. In view of recent experience at Berkeley with the one large still available, it is probable that there will be some changes in design to provide more convenient operation and less opportunity for contamination of the product by moist air.

The washing and recovery equipment has been largely designed and chosen by TEC. The purification of solutions proposed at present is a carbonate purification to remove iron, followed by precipitation of the tuballoy as TO_4.

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CHEMICAL ASPECTS OF THE BETA PROCESS

M.D. Kamen

1. General Considerations

The unique character of the beta process is manifested mainly in the close interplay or rigid chemical requirements with physical design of unit apparatus. As discussed in many previous reports, there must be provision for rapid recycling of unused alpha product, which is the only feed material. This in turn necessitates relative ease of recoverability of both dissipated charge and uncollected but processed material. In addition there is the dual requirement of minimal holdup in all chemical operations incident to the recycling process and avoidance of absolute loss of charge. The beta machines, which have been designed to meet these requirements, represent a compromise between the ideal which can be imagined and the practical which can be attained due to limitations of time and material.

To begin with, we may review the background for the decision as to the size of beta units. It appeared desirable at first to attain rapidity of cycling by constructing small units (i.e. machines operating on a one-foot I path radius or less) since it thereby became possible to construct a mechanical system which could be kept closed during the washing operations and would require a minimum of complicated mechanical equipment such as would be needed for disassembling any machine of a size such as R1. In the extreme, one could visualize extremely small units which could be handled by a single chemist such as a test tube operation. These could be

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made all glass and with a minimum of corrodable parts. This dream was shattered by consideration of the difficulties arising in collecting the separated beams of PX^{9} and Q^{9}. It only appeared practicable to collect the F^{9} on a circle of two feet or more, automatically setting the size of the beta machines at something intermediate between the 37" and the large tanks on the hill. A number of suggestions for collecting the PX separately at the pocket and the Q back in the middle of the outfit were advanced but nothing came of this. The main objection to the large outfits was the vast quantity of tank wash required to adequately clean such machines, resulting in prohibitively large evaporation installations. This objection was obviated by the realization that the major portion of the charge distilled out in any run could be condensed in the immediate vicinity of the H by a suitably constructed gunk catcher, so that, by confining the daily cycle to the bulk of the unchanged TA collected in this fashion, small amounts of wash could be used. This combined the advantages of the small outfits with those accruing to the big machines. The inclusion of a liner to shield the D^{9} wall of all material escaping from the gunk catcher , and adequate baffling in the pump leads completed the picture.

A suggestion that the beta machines be provided with mechanisms which continuously, or at frequent intervals, scraped the gunk catcher and returned unused TA to the H was made. This idea appealed to all as a means of increasing

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the speed of the daily cycle and reducing holdup. The difficulty in designing such a gadget was soon found to be such that it was not felt wise to risk loss of time on the part of the already overtaxed design and engineering staff in an attempt to develop a continuous cycling beta machine.

The experience of the laboratory thus led to a formulation of the beta machine as a modification of the unit used in the alpha process with the following recommendations:

1. The M* was to be insulated and removable along with the gunk catcher as one unit.
2. The gunk catcher was to be designed to cover as completely as possible the entire M region and extend far enough out into the B to condense 80 to 90 per cent of the ThI_4 vapor volatilized.
3. The use of corrodable materials such as copper, silver, brass, and others found by the laboratory to be attacked excessively by the ThI_4 vapor, was to be minimized or avoided entirely. The use of stainless steel, graphite, nickel, etc., was recommended.

   Note: To simplify and accelerate chemical procedures, it is proposed that the machines be made up of as few chemical elements as possible. Specifically, stainless steel is recommended for M materials and gunk catcher, with nickel-plated copper where good conductivity is absolutely essential. Graphite,

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which causes no complications in chemical cycling, could be used for slits and heat resistant surfaces. The liner is to be made of stainless, with the receivers of stainless and graphite wherever the heat is a problem. Difficulties in the casting of stainless steel have been cited as objections to this and substitutes such as Hirenite mentioned. The experience of the X beta X¹⁰ plant is required to settle on the final composition of the H. But in any case, by confining the impurities possible to a relatively limited number of elements, a fast chemical cycle can be constructed. It does not require many of the elements ordinarily encountered in B construction to render the chemical cycle for recovery of pure ? extremely difficult, even if there is plenty of time for purification. By the use of materials not easily corroded, minimal amounts of impurities will occur in the hot water B wash, and thus a solution adapted to rapid chemical treatment may be obtained.

4. Separate X's for PX and Q beams were to be furnished and their construction was to be such that no contamination of the PX¹⁰ pocket, either inside or outside, by sputtered Q could occur. It was considered desirable that the PX pocket be water-tight so that minimal amounts of acid could be used for cleaning pockets of collected PX.

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5. The pump manifold was to be moved to a point far from the M region and so baffled that no vapor could get by a direct path to the pumps. The TA_4 was to be a source material so that condensation occurred near the M, whether the J was running or not. The use of TC_6 was abandoned early in considerations of the beta process, because of the relatively high vapor pressure of the TC_6 at ordinary temperatures, as well as the risk of loss down the pump line if the J failed and the hot M was left spewing out undecomposed TC_6 into the B chamber. (It may be interpolated here that there still exists the possibility of using the TC_6^{35} in the beta process if an adequate chemical procedure can be devised for the direct synthesis of TC_6. The major objection to the use of TC_6 in the beta process at present is that there is bound to be a larger holdup in the chemical cycling since twice as much material must be cycled per gram of TC_6. A direct method of synthesis will make it possible to consider again the use of TC_6 in the beta machines. In this case, the pump line will have to be designed to catch all TC_6 vapor, possibly by the use of a hot copper grid followed by a liquid air trap. In this connection, the writer would urge the use of mercury diffusion pumps, since recovery of TC_6 from mercury would be very simple, merely requiring cycling of dirty mercury through dilute hydrochloric acid.)

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6. It is our recommendation that the beta chemistry cycling be carried out in a location as close to the beta machines as possible. It is not considered wise to transfer recovered chemical material from the machines any large distance to the region in which the chemical cycling is carried out. The beta chemistry is carried out in a limited space, preferably in one room. The apparatus and procedure contemplated can be adapted easily to fill this requirement. A separate pump for the beta chemistry room should be provided so that in event of spillage the beta material can be recovered in not too excessive volume by washing down into the beta sump. It is not recommended that the beta chemistry be spread over a large region. The points listed above will be treated at somewhat more length in the appropriate sections of the discussion which follows.

2. Beta Chemical Cycle

The main beta chemical cycle consists of the following sequence of operations:

1. Daily removal of M and gunk catcher and, less frequently, of the liner.
2. Daily servicing of alpha PX E's and separate treatment in similar fashion of beta PX E's. Isotopic analysis of the product.

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3. Hot water and steam wash of M and gunk catcher daily, followed at less frequent intervals by acid wash.

4. Evaporation of wash solutions after oxidation of T (with peroxide).

5. Purification of concentrate. This involves precipitation and filtration of any one of a number of T compounds. All discarded filtrates and residues are saved for salvage operation.

6. Preparation of oxides from separated and purified T precipitate.

7. Chlorination of oxide.

8. Salvage operations on condensate from evaporation, etc.

We will take up the steps in the order indicated.

a. Servicing of E's and Isotopic Analysis

The T deposit which appears on the E is made up mainly of metal and water-insoluble material, and one can expect a relatively large fraction of the total deposit processed to be impurities. The material best suited for use here is stainless steel (18-8). This is because T shows no marked tendency to bond strongly to stainless steel surfaces and because in the subsequent chemistry iron, nickel, and chromium present no great difficulty in the T purification. Moreover, as will be seen, the ions mentioned are the same as those occuring in the water wash of the main cycle, so that the purification methods can be similar. It is strongly recommended that the stainless used contain no more than

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one per cent molybdenum in addition to the usual iron, nickel, and chromium content and traces of other metals. Carbon is recommended for surfaces subjected to intense heating. Under no circumstances should plating be resorted to, as the F will sputter the plating and expose the main body of the metal. Furthermore, copper binds T firmly. If any large surface of copper is exposed, much of the T will be recovered only at the expense of dissolving practically all the copper. By treatment with hot 2N nitric acid, the T deposit can be recovered quantitatively from stainless, leaving a smooth, clean surface. In practice, it has been found that one hour of treatment with the hot acid removes better than 95 per cent of the T from the K's. It is also possible that some sort of spray wash will prove advantageous. We have had no experience with this type of procedure, however. It will be necessary to ash graphite at infrequent intervals to recover a small amount of T, which is driven deep into the carbon, due to the rather porous structure. T metal scattered from hot carbon surfaces appears to deposit in an insoluble form; probably a graphitized surface deposit over and in the T inactivates the metal as far as solution in acid is concerned. An ignition of this deposit in oxygen renders it soluble in acid. Other materials which give quantitative removal are nickel, molybdenum, chromium, stainless steels through a wide range of composition, and tungsten. However, nickel is not suitable if nitric acid is used, since excessive amounts will dissolve it. Chromium offers no

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advantage over stainless and is hard to obtain in solid sheets. Tantalum is possible as a collector material but offers mechanical difficulties, besides cleaning less easily with acid, since it has a tendency to combine with metal at high temperatures. Most of the collection occurs on the bounce, as it were, the F initially hitting the E back surface and reflecting to the stainless sides of the E. However, the I's still possess relatively high energies, chemically speaking, so that tantalum, which ordinarily is unreactive at low temperatures, bonds the I stubbornly if used in the E. The use of hydrochloric and peroxide offers some advantage over nitrate in that no nitric acid is introduced into the chemical operation of the main cycle thus complicating the evaporation scheme. It has been our experience that rather extensive solution of the E metal occurs with the concentration required (about 5 per cent HCl and peroxide). More experiments on this point can be done, however. Persulfuric acid is also effective in much the same way, but adds complexity to the chemical cycle. It is recommended that nitric be used as a wash liquid. The I & impurities could be precipitated with ammonia and the precipitate added to the main cycle after thorough washing, the filtrate going to salvage. Before addition to the main chemical cycle, a sample should be taken for isotopic analysis, which can be done by the spectrometer method, or by the counting method. Both are necessary in some cases as checks. The spectrometer method requires preparation of the UF_6, which is itself a

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good purification procedure. The process has been worked out completely at Columbia University and is based on the use of cobalt trifluoride. The method of preparation of the oxide for the reaction is not critical. A large excess of cobalt trifluoride is mixed with T_3O_8 and heated in vacuo to about 330°C. The required amount of T_3O_8 at present is fifteen milligrams or more. A risk is entailed in that excessive loss of T can occur if there is breakage during a reaction. The TF_6 need only be formed in low yield (about 10 per cent) and can be used without further purification after being condensed in a sample bulb with liquid air. The careful purification of the oxide is not necessary. Even if metals are present which give volatile fluorides, no complication is introduced, since TF_5^+ is the ion used for the analysis. Only a few milligrams of it are actually used, the bulk of the charge being retained back of the needle valve used to introduce the vapor into the spectrometer. The accuracy of the method is well beyond that required for the analysis. The counting method involves less risk in that no volatile compound is used, but elaborate purification is required since one of the quantities needed is total amount of T present in the sample. The acid wash aliquot is concentrated and T is precipitated and given a preliminary carbonate purification. Any one of a number of procedures can be used from this point, and, if an accurate determination is not wanted, quantitative recovery of it as pure TO_2 is not required. The final step involves TF_4 or TO_2

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onto a platinum or brass plate, in an alkaline medium, obtaining a homogeneous film containing up to several milligrams T per square centimeter. Details of this method are available in the Radiation Laboratory reports. It seems best to have individual treatment of B banks, rather than some assembly line process, since the number of banks to be processed per day is small (up to seven an hour). We have tried electrolytic procedures (both AC and DC) as a means of removing T from B's but find the process too slow and unnecessarily complicated compared to the simple acid wash. Furthermore, no less impurity is introduced into the B wash by this procedure. It may be pointed out, however, that electrolytic removal of the T in salvage procedures on the B material is a possibility. The treatment of the beta B's is similar in all respects. In all the operations here as elsewhere in the beta process, extreme care must be taken to prevent loss by spillage, spattering, etc. All operations should be conducted over vessels or flooring adequate to recover any liquid lost by spillage.

b. Treatment of the M and Gunk Catcher

In the beta process, about 90 per cent of the Th is volatilized practically unchanged from the M region, and condenses to a large extent nearby on the cool surfaces provided by gunk catcher and portions of M. About 2 per cent of this charge, or less, goes to metal and into water-insoluble portions. The first operation, therefore, consists in removing these parts in such a manner as to avoid any loss of T & deposited. There are several safeguards [illegible]

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the gunk catcher itself acts as a protective device against dropping the deposit from M. Immediately after removal, M and the liner are wheeled to the washing region. Alternatively a stainless steel vessel, in which steam and wash operations is to be performed, can be clamped in water-tight fashion over M and the gunk catcher. Steam and hot water may be introduced into this vessel and a quiescent treatment used, or M and the gunk catcher may be dunked in a series of tanks containing hot water (up to 90°C), soaking for periods up to half an hour. By far the major portion of Th will be recovered in the first dunking. The other rinses may be used for operations on succeeding B wash. Before removing the stainless steel washing box, if such is used, the apparatus should be dried thoroughly in a stream of hot air which is aspirated through the box and then through a filter cartridge for de-entraining the vapor. This cartridge can go at infrequent intervals to salvage. After the water rinse, the vessel may be filled with hot 2N nitric acid and allowed to soak for fifteen minutes, or alternatively, the source may be sprayed with hot 2N nitric acid and soaked in separate tanks containing the acid. It is essential that no nitric acid be mixed with the chloride wash. The combined acid wash and water rinse that follows is stored for use again on the next source, the acid makeup to be made on subsequent rinses so as to maintain the acidity at about 2N. The reason that nitric acid must be excluded from the main water wash is that otherwise the concentrate resulting from the

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evaporation procedure will build up large quantities of nitric acid. This will mean excessive salt formation during the neutralizations required for purification of T and this is undesirable for quantitative precipitation of T. If hydrochloric + peroxide can be substituted for nitric acid, the nuisance of having to keep two separate lines running through the beta cycle can be avoided. The recent work here and at Pantex seems to indicate that this is a good prospect. Stainless steel apparatus would be required for the nitric wash and tantalum ware for the evaporator handling the Th.

The liner is expected to catch about 5 per cent of the distilled charge each run. It will probably have to be treated every five or ten days, although provisions have been made to handle it every day. The same sequence of washing operations should be employed with the following additions:

The water washing rinse should be stored and used several times until T content builds up sufficiently to add to the main cycle. The nitric wash on the H can be recycled over the liner. The technique will involve enclosing the liner in a tank equipped with a spray system so that the liner can be washed down with a minimum volume. It is not advised to use quiescent treatment as in the H. The H, gunk catcher and liner are removed after washing, to be serviced preparatory to the next run. Whatever parts are removed are scraped and the scrapings carefully saved to be turned over to salvage.

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9. Evaporation

The thorniest problem in the beta cycle is the evaporation of the T wash solutions to a volume and concentration required for adequate chemical processing. In the beta plant at equilibrium there are expected to be some 25,000 pounds of solution containing approximately forty pounds of T coming out of the beta machines each day. This must be concentrated to about 5 per cent in T daily. In other words, a thousand pounds per hour must be evaporated to thirty-three pounds. The final hourly concentrate will contain about 1.6 pounds of T. These figures are approximate and plant procedure may change, but they have been used as the basis here for design of the evaporator units. There must be no, or minimal, holdup and absolutely no loss by entrainment or leaks. The choice is confined automatically to metal evaporator units, and, since the solutions run high in chloride, tantalum must be used. It is most fortunate that the problem of chloride corrosion has been neatly solved by the appearance of tantalum in the chemical and industrial field, as otherwise it would be most difficult, if not impossible, to construct an adequate evaporator for our purpose. The arguments and details of the evaporator construction and procedure as recommended by the Radiation Laboratory are discussed by Mr. R. Q. Boyer in a separate report. We need only mention here that there will be two effects. The first is to be a standard vertical tube evaporator to run on a ratio of 3/1, and the second is to be a high-speed,

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bayonet-type, partially submerged, to reach a final concentration of 10/1. The over-all concentration factor is 30. There will be no submerged valves. All parts exposed to strong hydrochloric will be tantalum and the control will be by means of plungers operated through a seal high above in the vapor phase. The riser will be glass lined and fixed to the tantalum unit be a tantalum seal and flange. A de-entrainment column packed with glass saddles and designed to reduce entrainment by at least a factor of a thousand follows the riser. There is, finally, a condenser, also glass lined, which condenses the vapor to storage. The de-entrainment separator and condenser can be affixed to each effect. The concentration of hydrochloric appearing in the final concentrate is debatable at the moment, but is not expected to exceed 10^{-6}M. This is in contrast to the nitric acid line, which will be handling concentrated nitric in the condenser. Units capable of handling five hundred, one thousand, two thousand, and four thousand pounds a day should be constructed to permit flexibility in the flow of operations, especially in the build-up period. The operation is batch-wise to reduce holdup, which occurs mainly in the final effect. The problem of treatment of the condensate will be taken up in the discussion on salvage operations. Before evaporation, the tank wash must be filtered. A coarse filter will suffice. The transfer of the concentrate to the chemical treatment vessels is expected to be done with syphon attachments. The

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availability of a good tantalum valve will change this, however, as well as the method of control of feed to the two effects.

The above procedure is based on experimental models which have been demonstrated to be workable. At the present time further research is being carried on to see in what way the evaporators can be further modified and simplified. It may be mentioned here that the use of liquid-in-tube types may supersede the bayonet type for the second effect if experiments now under way prove successful. Reference is made to the report by Mr. Boyer for full details.

d. Methods for Purification of Concentrate

The methods of purification are determined by the nature and the amount of the impurities present in the tank wash. There is no simple method available which satisfies both criteria of minimal number of chemical manipulations and quantitative recovery of T free of impurity. We have investigated, in more or less detail, a large number of possible schemes and will describe here those which appear suitable for treatment of tank wash. We have been guided here by the expectation that the main contaminant will be iron, with smaller amounts of nickel and chromium. All insoluble tungsten, carbon, etc., which has been removed in the filtration preceding the evaporation is saved for salvage. There may occur traces of copper, molybdenum, etc., but so long as they remain in trace amounts, they will not complicate procedures outlined below:

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(1) Electrolytic Procedure. This is perhaps the only universally adaptable method for all types of wash solutions encountered. Practically all metallic ions can be separated by this procedure, based on the use of a mercury cathode and rotating inert anode of platinum or carbon. The electrolysis is done in sulfuric acid solutions (1/10 to 1N). Under the proper conditions of current flow and voltage, metals such as iron, nickel, chromium, molybdenum, vanadium, zinc, sodium, etc., are quantitatively plated out and into the mercury. All the tubanyl is reduced to tubonous in the course of electrolysis. We have not extended this method to the processing of large amounts of T (that is, one-pound batches). A typical unit would require a stainless envelope water jacket with insulated flange top, fitted with vapor-tight liquid seals for stirrers, and vapor de-entrainment, and a series of stainless leads and valves for drawing off the tubalious solutions and the dirty mercury after electrolysis. It has been shown in tracer experiments that, when the acid is kept above one tenth normal, no detectable T can be found in or on the mercury, even when enormous current densities (up to twelve amperes per square centimeter) are employed. The usual conditions for electrode separation of three to five centimeters are six to eight volts, and three tenths to five tenths amperes per square centimeter. The time required depends on the total amount of impurities present. For a cell holding about five hundred centimeters of 5 per cent T and equivalent amounts of iron, nickel, etc., about

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six to eight hours are required at present to reduce the impurities to a point where the solution is considered pure enough. This can be anything less than one tenth to five tenths per cent in iron, etc. A design of cells with large surface and more adequate cooling should cut the time considerably. If a little impurity is present, the total time will be much less. It is necessary to run the electrolysis in sulfuric acid solution. Nitrates or chlorides interfere by lowering the current efficiency and introducing undesirable secondary reactions such as re-oxidation of the impurities plating out on the mercury. Hence, a conversion to sulfate is required, which means an ammonia precipitation and a re-solution in sulfuric acid. The solutions can be drawn off with the current on, although we have found that no appreciable re-solution of the impurities occurs if the current is left off during the period that the solution is being transferred. An oxygen-free atmosphere helps here. The dirty mercury is cleaned by passing through dilute acid (hydrochloric acid), the acid being thrown back into the main cycle. There is a detailed report (Chem S 16) on this method available in the Radiation Laboratory.

We have not carried out this electrochemical procedure on a pilot plant scale but would estimate that a generator capable of about fifteen hundred amperes output at ten to twelve volts would be sufficient to handle the beta plant at full operation. Such a generator has in fact been ordered for the site.

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(2) pH Control Method. This method depends on the relative insolubility of tubalious hydroxide at pH of about four, as compared to the hydroxides of ferrous, nickelous, chromous, etc. An adequate Beckman control unit with reliable redox electrode is required. The reduction of the ionic constituents of the B wash is accomplished with hyposulfite (ammonium or sodium salt). We have worked with the ammonium salt mainly. At one time we thought it desirable to get rid of excess salts in salvage by ignition. Actually this is not contemplated now as there is too much danger of losing T when igniting off ammonium salts. It should be easier to work with the sodium salt, although at the pH used there is relatively little difference in stability. It is essential that the hyposulfite be prepared with as little sulfite present as possible, although a mole to mole ratio of sulfite to hyposulfite can be tolerated. Excess sulfite results in loss of the T by re-solution. A procedure has been worked out in detail and at present recoveries of 99.9+ in T are obtained regularly with extremely high purities. In most cases no iron has been detected in these tubalious precipitates by any of the usual colorimetric tests. The filtrate has been examined, using tracer techniques, so that this high percentage recovery has been established and checked by means of a procedure which is reliable for extremely small amounts of T. Formaldehyde is used at the end of the titration with hyposulfite to remove the excess sulfite which is formed in the reaction. The occasional

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appearance of a large amount of insoluble organic material, presumably hexamethylene tetramine, etc., has been noted in the first precipitation. The use of the sodium salt should obviate this difficulty. At concentration of T below one per cent the formaldehyde is not required in such amounts as to introduce this complication. This pH procedure has the advantage that the only manipulations are precipitation and filtration. We have settled definitely on the avoidance of centrifuging operations, as these involve too much risk of loss. The precipitate as formed contains from 2 to 5 per cent impurity as sulfur, after a mild drying at about 120°C. If it is desired to remove this sulfur, the filtrate can be dissolved off the filter in HCl and reprecipitated as a tubalious hydroxide with alkali. The precipitation is done in the cold with acid pH and in the hot with alkali. The precipitate obtained in either case, that is, either in acid or base under these conditions, is readily filterable in contrast to most others obtained by the methods that are available. It is necessary to avoid oxidation of the tubalious hydroxide during these operations, so there should be a stream of nitrogen run over the filter cake during the solution operation. It is possible, however, that the first precipitate, even though containing the sulfur impurity, will prove adequate as starting material for making the chloride. This point is now being investigated. The sequence of operations involves transferring the oxide from the filter cake to the chlorination reactor. We have designed a filter unit.

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consisting of a tantalum perforated plate backing a Vycor filter cake. Precipitations are carried out in the tantalum-lined iron cassette which is provided with pipes for cooling and heating. The filter cake is then clamped on and filtration carried out under steam pressure. The steam is introduced through a pipe which is part of the filter unit. There should be a separate pressure unit for production of pure steam available in the beta plant. After filtration, the walls are washed down with steam. The cake is removed, the precipitate dried and transferred in the Vycor to the chlorination vessel. It may prove necessary in the end to use a filter material finer than is available as Vycor cake. This will be the case if a purification based on a dithallate or peroxide is decided upon. A sintered glass disc can be used in this eventuality. The diameter of such fritted glass discs is not large enough in available models, so a conical adapter may be required. The necessity for pressure filtration will involve designing sufficiently strong backing to use such fritted discs. Possibly the use of moderate pressures well below the tolerance limits on fritted discs will be sufficient, especially if the disc diameter is not large. The use of paper or organic filter media is not recommended. Likewise thick filter cakes involving large holdup are to be avoided. The technique for transfer depends on the method of chlorination proposed. If the transfer is made in such a way that the bulk of the precipitate is transferred off the Vycor, the residue on the Vycor is turned

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back either to salvage or to the main cycle.

The total volume of the cassette up to the filter unit is estimated to be ten gallons. There should be a cradle provided to handle the cassette.

(3) Peroxide Method. Tuballoy peroxide is insoluble in acid solutions in which the chloride concentration is not too high. This fact can be utilized to separate T from the contaminants to be encountered. Unfortunately, in the presence of a large amount of iron, excessive decomposition of the hydrogen peroxide takes place as it is added to the solution. Consequently the iron must be tied up in some kind of a complex with acetate, lactate, etc. Furthermore, the precipitation must be carried out in the cold, since the complex decomposes on heating. There is some solubility of the tuballoy peroxide in the concentration used to prevent co-precipitation of iron, nickel, and chromium. However, this method is suitable especially in conjunction with the standard carbonate method for purification of samples for isotopic analysis. The extension of this method to the purification of B wash where little iron is encountered should be considered seriously. The major objection at present is that the peroxide is difficult to filter when precipitated in the cold. If precipitation is done in the hot, as is possible with the low amount of iron, the method may conceivably be used. Reference should be made to the reports by Dr. J.I. Larsen for full details.

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(A) Carbonate Method. This is the classical, and worst, procedure possible. The method depends on the solubility of tubanyl in excess carbonate, while iron, nickel, and chromium precipitate as hydroxides or carbonates. In the presence of large amounts of iron at least three or four precipitations of the iron hydroxide are required to quantitatively recover all the P. Furthermore, a precipitation is tricky if an easily filterable hydroxide is to be obtained. We usually use this method in conjunction with the centrifuge, which, however, is not advisable for the main beta cycle chemical operations. If a very small amount of iron is present, this method is one of the easiest. In fact, it may be mentioned here that, if the impurities in the tank wash are low, there will be no problem involved in purifying the tank wash, as any one of these methods will be equally adaptable. Full details on this procedure are available in Radiation Laboratory research notes.

(5) Cyanide Method. A possible alternative method to the complex acetate and lactate is the use of cyanide. In this procedure the potassium cyanide is added to the solution made slightly acid with HCl. No oxidation is resorted to here. After excess cyanide has been added, the solution is neutralized with ammonia and ammonium dihydrate is precipitated. Iron, nickel and chromium in the oxidized states are held up as complex ions. This method needs a good deal more work but is mentioned here as a possibility.

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(6) Ether Method. T can be extracted from a mixture of nitrates by successive extractions with acidified ether (6M in HNO_3) or, alternatively, if iron is a major contaminant, the chloride solution can be extracted with ether saturated with 6M hydrochloric acid, which removes the iron.

(7) Chromatographic Separation. We have had some experience here with the preferential absorption of T, iron and nickel on various materials such as aluminum, silica and ion exchange resins such as Amberlite. The major disadvantage is that for a preferential absorption, the columns must be developed slowly and a large holdup occurs. Complete absorption of all multivalent metal ions can be accomplished quickly using the Amberlite resin, and, in fact, this method serves as a basis for salvage operation. A two-foot bed of the grade of Amberlite known as IR-1 will reduce the concentration of T in the liquor processed by a factor of 10^{-5}. Thus a solution containing 1 mgm. T/liter after passage through the Amberlite column showed a final concentration of less than 10^{-5} mgm liter. The velocity of flow should not exceed 50 gallons/ft^2 per hour. The absorbent saturates at about 50 mgm T/gram absorbent. The solution must not be too acid (i.e. greater than 10^{-2}M), as H^+ ion is the most easily absorbed ion. Multivalent ions such as Fe^{+++}, Ni^{++}, UO_2^{++}, etc. are absorbed preferentially and completely in this type of resin, the Na^+ or NH_4^+ which is the cation activator being displaced. We have not had much experience in this laboratory with this type of absorbent,

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the figures given above being based on work done at the Metallurgical Laboratory in Chicago. We are investigating the ion exchange method now and checking the Chicago observations.

(8) Reduction Procedures. Reducing agents other than hyposulfite are available for converting the T present in the tank wash to tuballous. In particular, one can run the solution through an iron reductor. This is then followed by the usual pH control precipitation. There seems to be no advantage of this method over the method outlined under No. 2. And in addition there is the disadvantage that a large amount of holdup occurs in the reductor. However, for dilute solutions of T containing appreciable amounts of Fe, such a reductor could be used, followed by hydrolytic precipitation with pH control.

In all the operations outlined above there is some necessity for testing solutions and residues microanalytically. If a little T is present, mixed with large amounts of other ions, we would recommend electrolytically purifying solutions obtained from the residues, followed by either concentration by evaporation or, if the concentration of T is high enough, direct application of spot-test methods. For spot tests, there is available the usual ferricyanide test, as well as a number of others which have been worked on in this laboratory. The removal of excess salt before making the spot test could be accomplished by the ion-exchanger. The

various methods outlined above have been discussed in detail in the Radiation Laboratory reports and reference should be made to them for methods of procedure.

a. Chlorination

In the report on the alpha process Dr. Carter has described the available methods for chlorination of the oxide. The same remarks apply to the chlorination of the oxide in the beta cycle, with the added provision that there be no loss and little holdup of F during the chlorination procedure. The process, which recommends itself, is some modification of the vapor phase method. The use of the Davis reactor with positive pressure seals and electrostatic precipitator at the outlet and is contemplated here. Research is now proceeding using tracer techniques to determine what, if any, loss occurs using the Davis reactor. The holdup is low, amounting only to the amount of oxide required to fill the tube of the continuous reactor. During the build-up period a very small reactor will be required. At equilibrium a four-inch reactor with total holdup of about three hundred grams is in prospect. A further advantage of this type of equipment is that transfer of the chlorides to bottles is accomplished simply, with little or no risk of loss, as the outlet of the chlorination tube leads directly into the F bottle. Except for outgassing the product it is not recommended that the material be redistilled. The use of the liquid phase reactor is at present distinctly less highly regarded. The product made

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is the pentachloride, TCl_5, and this has to be broken down to TCl_4 before opening a reactor. Adequate provision for stirring without loss of T must be made. There must be complete reaction of the charge. It also appears that a large excess of TCl_5 is needed as catalyst, leading to excessive holdup. Dr. Kraus and his group at Brown University are investigating this reaction as a possible beta chlorination procedure. It is possible that by the addition of reducing impurities such as sulfur, water, etc., in minute amounts, the reactor can be made to produce only TCl_4 and thus cut down loss during the reaction as TCl_5. This point still has to be investigated. There is a procedure which is best suited from a standpoint of convenience for the beta process. This is the one based on the direct reaction of liquid sulfur chloride with any or all tuballoy oxides. No TCl_5 is formed and the reaction occurs at room pressure at the boiling point of sulfur chloride. Unfortunately the reaction is slow and it is not advised at the present time for the production of the chloride. The product obtained has to be purified of sulfur and we have accomplished this in a variety of ways. One way involves the extraction of excess sulfur with an excess of sulfur chloride, followed by carbon tetrachloride extraction and finally drying. The second involves subliming the sulfur away from the TCl_4 at a high temperature in vacuum. Both these operations can be carried out in a short time and yield a pure enough product. However, it is necessary to

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avoid loss in the subliming procedure and it is risky to use the procedure in which a large amount of valuable T4 is heated in vacue to remove sulfur. A breakage at this point would result in loss of a large quantity of beta feed. We have investigated a large number of other methods which offer possibilities for chlorination of the oxide. These are described in the Radiation Laboratory reports. None of these need be mentioned here as they appear to be of no special significance for the beta process. An important phase of the chlorination is the preparation of the oxide after its purification. Either TO_2 or TO_3 can be used in the vapor phase procedure, but TO_3 is required in the liquid phase reaction as it is presently operated. If necessary, T_3O_8 or TO_3 can be reduced easily with methane or hydrogen to TO_2. In the Davis reactor this would mean a preliminary treatment in a tube in series with the chlorination tube into which the freshly prepared TO_2 would be fed from the reduction chamber. Any kind of oxide, so long as it is reasonably dry, is useful for vapor phase reaction, but the oxide used in the Kraus reactor needs special treatment. All these points have been discussed in the report on the alpha process.

f. Salvage

This is an extremely important phase of beta operations. It turns the beta operation from a semi-quantitative system into a complete recovery scheme. We have to deal with several types of salvage operations. [illegible]

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with solutions very dilute in all metal constitutents, including T. This occurs in the condensate from the evaporation stage. In another phase of the operations, we are presented with solutions concentrated in metallic impurities such as iron, and dilute in T. The third type of residue is solid and covers all the tungsten and carbon and organic residues involved in evaporation from salvage operations on the previous solutions. We have a variety of methods available for handling these cases. The first is the carrier process. This method depends on the co-precipitation of micro amounts or small amounts of T, using as carrier calcium or lanthanum fluoride in acid solution. This method is recommended for either the first or second type of salvage operation. The second method involves electrolysis of the solutions and recovery of T from the electrolyzed solution. This method is recommended for the second type of salvage. The third method involves use of the Amberlite ion exchange resin. This material, which is sold as a sodium salt of a polymer of phenol, formaldehyde and sulfamic acid, is an extremely strong absorbent for all multivalent metal ions, including iron, nickel, chromium, and T. The solution to be processed is run through the resin and all metallic ions are removed as long as the solution is not too acid. Details of this procedure are to be discussed in a separate report. The fourth method is the ether extraction referred to in a previous section. A combination of any or all of these methods is certainly adequate for any of the situations which may arise in salvage.

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The major portion of the beta cycle requiring reprocessing in salvage is the evaporator condensate. If it is decided to recycle this back to the tank ~~was~~ (as now appears possible), the bulk of the salvage operations ceases to exist. If this is not the case, the condensate can be worked over with the lanthanum fluoride procedure. (One requires about 0.05 to 0.1 grams/liter of the lanthanum salt.) The fluoride is dissolved and used over and over until sufficient T is accumulated to make possible chemical recovery by the usual procedures (i.e. carbonate, ether, etc.). The Amberlite exchanger can also be used at this point in preference to the carrier procedure. All that is required is to run the solution through the exchanger, discarding the effluent after micro tests show no T to be present. A bed of IR-1 absorbent two feet deep and three feet in diameter is more than adequate to handle the beta salvage for this particular condensate. The elution is accomplished at regular intervals with a small quantity of concentrated (6M) H_2SO_4. The eluant should contain sufficient T to make it possible to recover the T by a straightforward macrochemical procedure. Otherwise, here as elsewhere T from the main beta cycle can be added as carrier. In fact it might be advantageous to reduce efficiency in the main beta operations and turn the salvage operation into merely a second-stage beta chemical procedure. The experience with X beta X will determine exactly how the salvage procedures should be worked in.

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The solid residues encountered here and there in the beta process will not in all likelihood contain significant quantities of T. It is expected that test-tube operations by one or two good analytical chemists will be adequate to handle this phase of salvage. The material most often encountered here will be graphite from B banks, slits, etc. It will be necessary to pulverize and ash the carbon after which the ash containing T is brought into solution and purified either separately or, if no appreciable impurity is present after the carbon has been burnt off, the T can be added directly to the main cycle. The filtrates from precipitates and reprecipitations are subject to the same general procedures as those outlined for the evaporator condensate. In general it would appear that the carrier technique and electrolytic cleanup will handle the bulk of salvage with the ion-exchange as a final clean-up in all solutions which have already been processed for salvage by one or the other of the methods described.

3. Bookkeeping in the Beta Process

At all times it will be desirable to have some notion of the amount of material in process. This requires accurate determinations of the T coming from the alpha E's. There will be a large uncertainty as to holdup in various parts of the beta cycle which will render it impossible to check on whether any absolute loss is occurring. The best that can be done is to make the entire cycle a closed system with nothing

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leaving the plant until the salvage crew has ascertained that no U is being thrown away. We have described a process in this report which emphasizes minimal holdup. We can estimate how much this holdup will be at equilibrium. In the U from alpha, no more than 0.1 per cent of the daily output will be held back because of the solubility of the precipitate. This is entirely negligible since the total makeup from the alpha stage is about 2 per cent of the circulating material in the beta process. That is, the holdup in the alpha U's, as far as the beta machines are concerned, is 1×10^{-3} of 2 per cent. The holdup in the beta machines will run between 5 and 10 per cent of the daily processed material. This should be by far the main chemical holdup. Most of this is recovered at intervals with the acid wash. The chemical holdup in the entire evaporator sequence is no more than the contents of one U bottle (i.e. one pound). Actually about 0.1 lb. holdup is expected here. This is 0.2 per cent of the daily process. Holdup in the connector and filter unit is negligible (less than 10^{-2} of evaporator holdup). The large chlorination reactor will require a holdup of about 1/2 to 1 pound. This is the most likely place for the main chemical holdup after the wash operation. In any case it can be seen that the chemical cycle will contribute very little to the total beta holdup which is encountered, mainly because of the low process factor in the beta machines; forty pounds of beta feed are required in

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daily circulation to maintain full production of beta product and of this no more than two to four pounds extra will be introduced by the necessity for chemically recycling the feed.

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3	Monthly Report - Chemical Group UCRL; April 1943; File R.L.4.5.8 (Chem. M-8).	Y-12 Operations Classified Files
4	TEC History - 1943; 19 June 1944	Y-12 Operations Classified Files
5	Report Chem. S-500; Evaporation of Beta Gunk Wash Solution; 6 May 1943	Tennessee Eastman Library - Y-12
6	~~Reference to be inserted by District Not available to author~~	~~This reference is not available in District Office (See Pg. 2.1) Suggest search in Washington Office.~~
7	Contract No. W-7405-eng-48 University of California 20 April 1943	District Contract Section Files
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APPENDIX "B"

GLOSSARY

Alpha. - Code name given to the plant separation process which charges normal UCl_4 into mass spectrograph and produces enriched uranium with a concentration of 10% U-235.

Alpha I refers to the first accepted spectrograph design (See App. A6). Alpha II refers to a later more elaborate spectrograph design (See App. A6).

Beta. - Code name given to the plant separation process which charges enriched UCl_4 obtained from Alpha or the Gas Diffusion Plants into a mass spectrograph and produces nearly pure uranium 235.

Charge Material. - A uranium salt, uranium tetrachloride, which is charged into the mass spectrograph to be volatilized by heating, ionized and accelerated through the magnetic field of the spectrograph.

Cyclotron. - A machine consisting of a powerful magnet and special high voltage equipment which is used to produce atomic or subatomic particles.

Enriched Material. - Uranium salts or uranium metal which contains more than the natural amount of isotopes U-235, that is, more than 0.7%.

Isotope. - Each element is made up of atoms chemically distinguishable from the atoms of every other element. However, most elements, themselves, are made up of two or more forms of atoms which

have slightly different masses. These atoms of differing mass are called isotopes of the element.

Liners. - Metal enclosures attached to a frame work supported from face plate for the mass spectrograph ion beams to prevent loss of uranium sublimation into the vacuum tank or bin. Appendix A6 shows a picture of an Alpha I unit. An Alpha I liner is a closed metal container, Semi-circular or D shaped like the open part of the unit shown in A6. Alpha II did not have liners.

Liquid Phase. - Methods of preparing UCl_4 in which UO_3 is charged to a reactor together with a liquid chlorinating agent such as CCl_4. The reaction is brought about by heating the mixture, usually under pressure.

Mass Spectrograph. - An instrument used to separate isotopes of an element by ionizing and accelerating them in an electrical field so that the heavy isotopes follow a slightly different path than the light isotopes.

Space Charge Limitation. - A phenomenon occurring in vacuum tubes and other electronic equipment when newly formed ions are accelerated by an electric field. These ions tend to repel one another because of their like charge, and hence retard the ion flow.

Uranium 235. - Atoms of uranium whose mass, compared to the mass of the hydrogen atom, is 235 units. Naturally occurs as about 0.7% of normal uranium.

Uranium 237. - A radioactive uranium isotope, not naturally occurring, which is produced by bombarding normal uranium with neutrons.

Vapor Phase. - Method of preparing UCl_4 charge material by heating UO_3 in a tube and allowing hot vapor of CCl_4 or other chlorinating agent to flow over the UO_3.

XA Magnet. - Full scale model of the Alpha magnets which was constructed at UCRL for early studies.

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